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AUGER ELECTRON SPECTROSCOPY STUDY OF SURFACE SEGREGATION IN COPPER-ALUMINUM ALLOYS

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SUMMARY

Surface segregation of aluminum has been observed in copper-aluminum alloys by use of LEED and Auger Electron Spectroscopy studies. The alloys were solid solutions of aluminum in copper having compositions of 1, 5, and 10 atomic percent aluminum. All samples were single crystals oriented in the (111) direction. Surface concentrations five times that in the bulk were observed. LEED and characteristic loss data gave confirming evidence of surface composition changes. Surface concentration was found to increase with temperature on heating to 700^o C.

INTRODUCTION

Surface composition and contamination can affect surface properties. For example, changes in both conditions radically affect adhesive properties and hence frictional properties of surfaces (refs. 1 and 2). Also, on the basis of adhesion and low energy electron diffraction (LEED) studies, there is evidence of surface segregation in copper-aluminum alloys (ref. 1).

Surface and grain-boundary segregation have been studied in the past (refs. 3 and 4). Although a number of theoretical explanations have been proposed for observed behavior (refs. 3, 5, and 6), the question of a model for surface segregation is not settled. This results, in part, from the fact that previous experimental studies (refs. 3 and 5) have not been quantitative. With the advent of LEED and Auger electron spectroscopy (AES), new powerful tools for examining surface structure and composition are available for quantitatively examining surface segregation.

The present study uses LEED and AES for examining changes in surface composition and structure in copper-aluminum alloys. The objective of the study is to begin quantitative examinations of surface segregation in binary alloys.

The alloys studied were solid solutions of aluminum in copper having compositions of 1, 5, and 10 atomic percent. All studies were performed on single crystals that were oriented in the (111) direction. Effects of temperature on surface segregation were also observed. These alloy systems were chosen because evidence of surface segregation had already been observed (ref. 1). In addition to direct observation of surface composition change, the energy shifts of characteristic losses (refs. 7 to 9) with composition were observed (specifically, the first copper plasmon loss, refs. 8 and 9). Studies of characteristic losses may provide information on the electronic structure of alloys.

MATERIALS

The single crystals studied were cylinders varying from 0.6 to 0.8 centimeters in diameter and 0.6 to 0.8 centimeters in height. All crystals were oriented in the (111) direction. The samples were substitutional solid solutions with 1, 5, and 10 atomic percent aluminum in copper. The crystals were triple-zone refined, resulting in 10 ppm impurities. Pure aluminum and copper single crystals with the same purities and orientations were used as standards for the Auger and characteristic loss data. All samples were polished to 600 grit on metallurgical papers and then electropolished in orthophosphoric acid.

APPARATUS

The apparatus used for these studies is shown in figure 1. The copper-aluminum single crystals could be rotated, allowing both AES and LEED analysis and ion-bombardment cleaning of the crystal surface.

The crystals were supported by tantalum strips formed about their circumference. The supports could be resistance heated by a 100-ampere alternating-current power supply. The crystals were sputter cleaned by use of an ion-bombardment gun with high-purity argon as the sputtering agent.

The AES and LEED systems used are standard, available commercial equipment. A block diagram schematic of the equipment is shown in figure 1. The vacuum system consisted of sorption pumps, an ion pump, and a sublimation pump. When data were taken, system pressures were maintained at 2×10^{-10} torr as measured by a nude ion gage. Surface temperatures of crystals were measured with an infrared pyrometer and later checked with a Chromel-Alumel thermocouple.

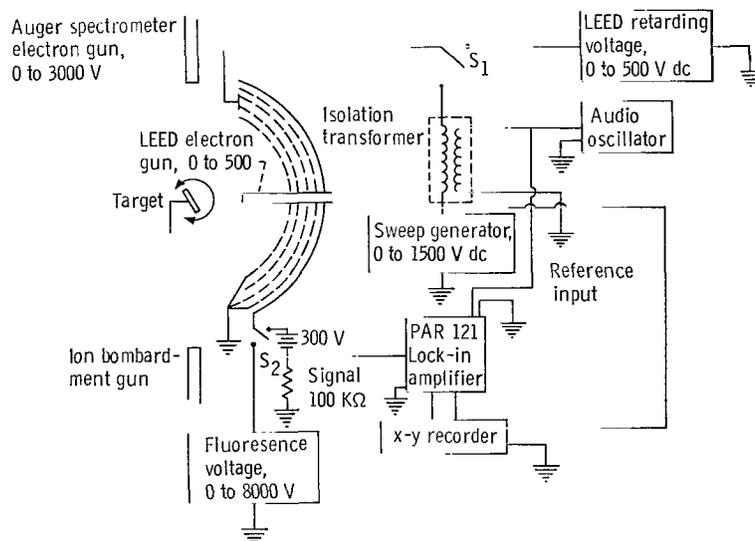
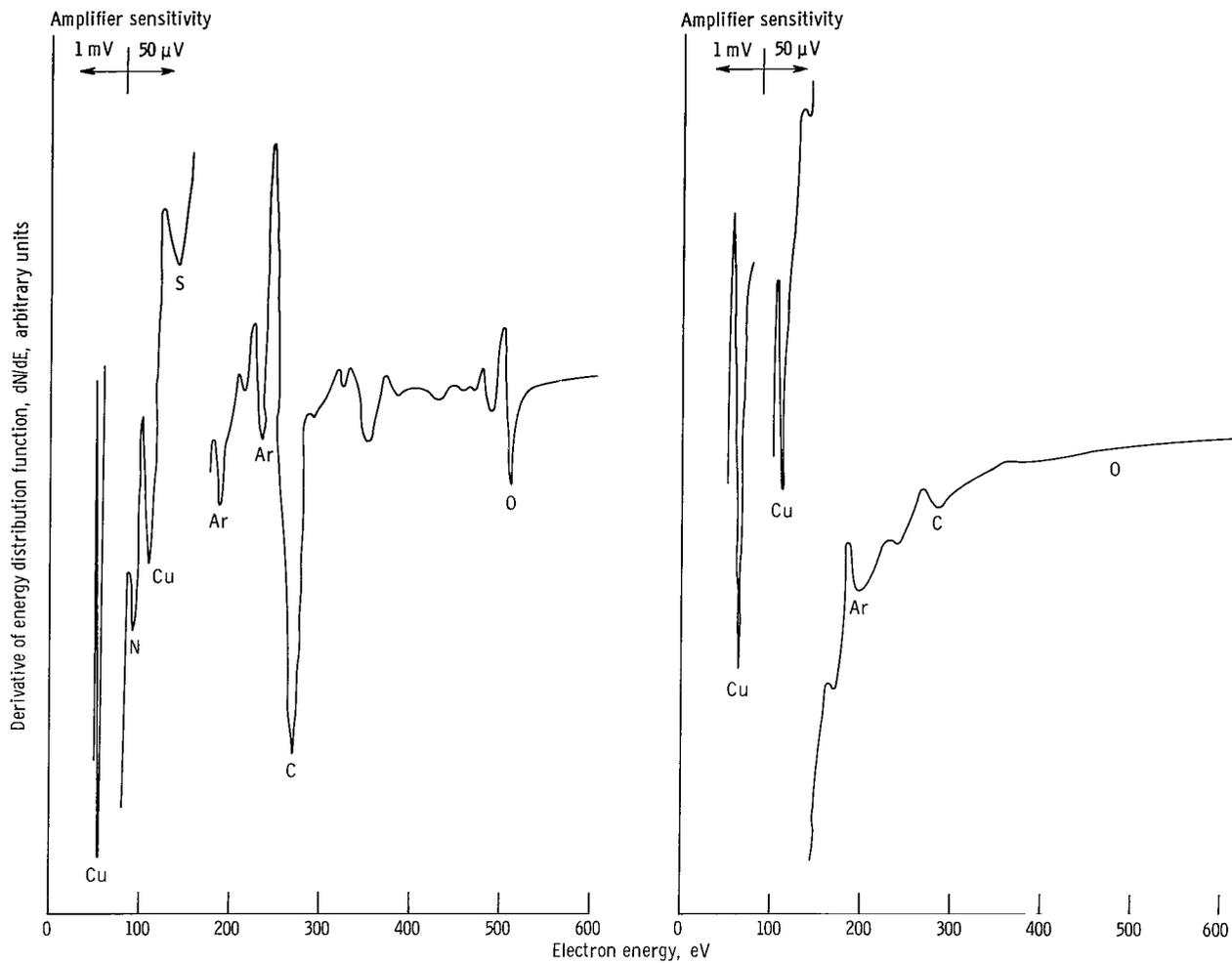


Figure 1. - LEED-AES apparatus.

EXPERIMENTAL

Pure copper (111) and aluminum (111) were studied in order to establish the location of the principal Auger and characteristic loss peaks of these metals. Although heating copper in hydrogen (ref. 2) and heating aluminum in vacuum (refs. 2 and 10) were effective surface cleaning techniques, argon ion-bombardment was found to be more expedient for surface cleaning. Repeated cycles of heating the crystals to approximately 500°C for 15 minutes and ion-bombardment for 1 hour at a 600-volt beam energy and a 5×10^{-6} -ampere-per-square-centimeter current density were adequate for surface cleaning. Crystals were annealed by heating them to 600°C after bombardment. The principal contaminants were carbon, oxygen, and sulfur. The samples were considered to be clean when the carbon, oxygen, and sulfur peaks appearing in the derivative (dN/dE) (ref. 11) of the secondary electron energy distribution curve were no longer observable for lock-in amplifier setting of 50-microvolt sensitivity, 10-volt peak-to-peak modulation voltage, 2500 electron-volt beam energy, 3-second time constant, and 25-volt-per-minute sweep speed. Figure 2 shows the effectiveness of sputtering for 1 hour at a 600-electron-volt beam energy and a 5×10^{-6} ampere-per-square-centimeter current density for surface cleaning. Breaks in the curve are a result of increasing sensitivity and zero shifting in order to fit traces on the page.

The location of the copper and aluminum Auger and characteristic loss peaks used in this study are shown in table I. These peak locations, which were determined in a separate study, agree with the results found in references 7, 9, 12, and 13. The high-



(a) Contaminated copper (111) surface.

(b) Same surface after argon ion bombardment at 600-electron-volt beam energy and 5×10^{-6} -ampere-per-square-centimeter current density for 1 hour.

Figure 2. - Derivative of energy distribution function as function of beam energy. Modulation voltage, 15 volts peak to peak.

TABLE I. - AUGER ENERGIES AND FIRST PLASMON PEAKS OF COPPER AND ALUMINUM

Material	Auger energies, eV	First plasmon peak, eV
Copper	57.3, 60.2	19.8
Aluminum	66.5	15

energy minimum in the Auger dN/dE curves were used to define peak location. The accompanying LEED patterns were a secondary indication that the surfaces were clean in that the spots had a high intensity and the background was dark.

The same cleaning procedure was used for the copper-aluminum alloys, that is, heating followed by argon ion bombardment. Because of the proximity of the copper and aluminum Auger peaks, it was necessary to use a 2-volt peak-to-peak modulation voltage and a 5-volt-per-minute sweep speed in order to obtain sufficient resolution. The lock-in amplifier was operated at both 20- and 10-microvolt sensitivities for these measurements.

The surface segregation data for the alloys were taken by first sputtering the crystals for periods of 15 hours or more at a beam energy of 600 electron volts and a current density of 5×10^{-6} amperes per square centimeter. Following the sputtering, the samples were heated in 100°C increments, at 30 minutes per increment, to a maximum temperature of 700°C . An AES trace was taken after sputtering and after each 30-minute heating. The surfaces were held at 600°C for as long as 64 hours in order to determine whether any further increase in surface concentration would occur. Each AES trace was repeated at least seven times to establish a standard deviation for the measurements. The LEED pattern was observed after each treatment.

Characteristic loss data were observed following sputtering and heating at the maximum temperature. For these data the direct-energy distribution function $N(E)$ (ref. 14) shown in figure 3 was used. An electron beam energy of 300 electron volts was used

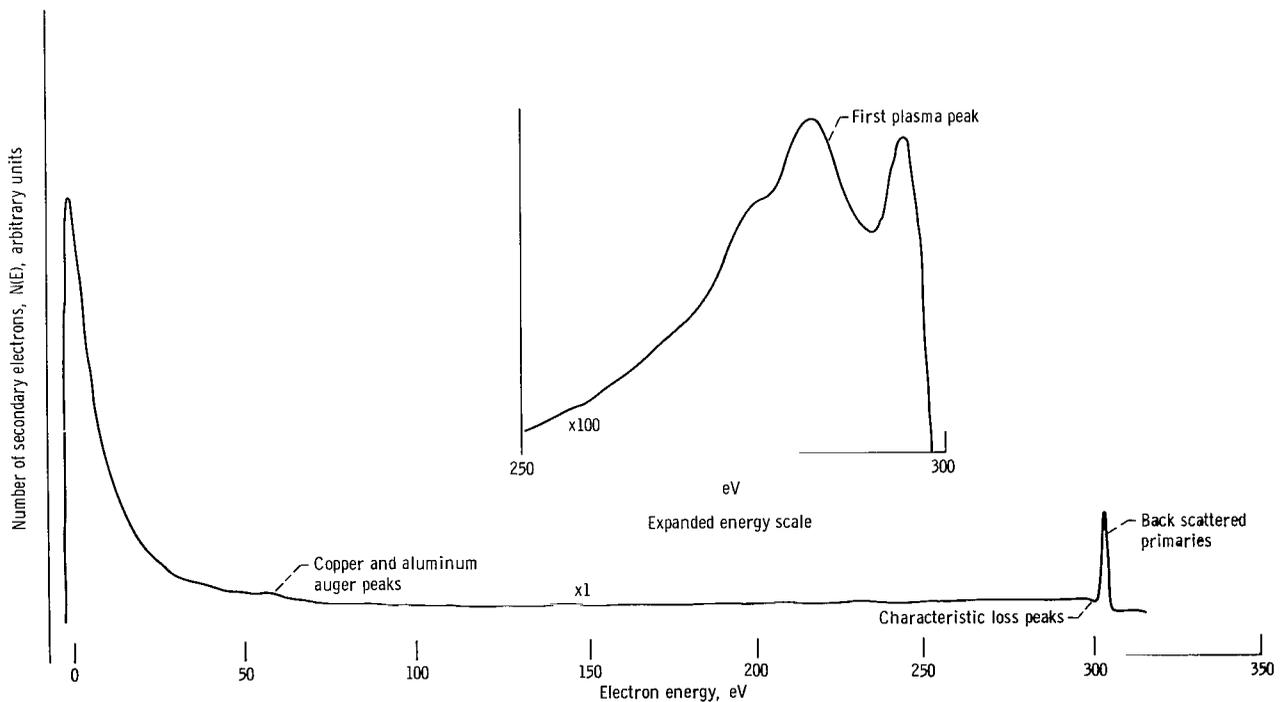
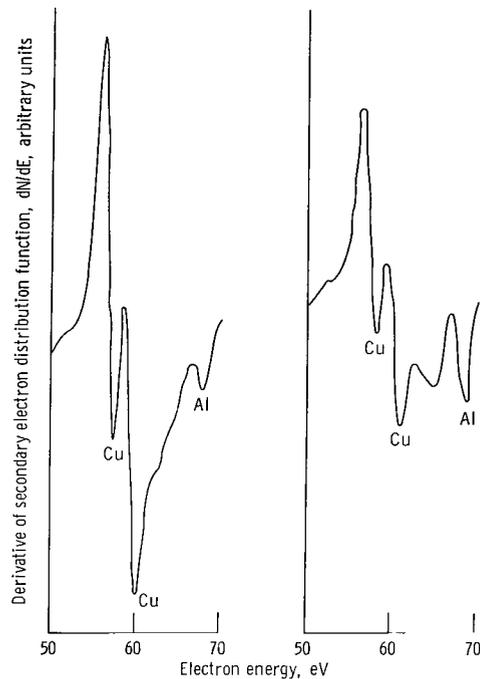


Figure 3. - Secondary electron energy distribution for copper - 10-percent aluminum alloy.



(a) After argon ion bombardment for 15 hours at current density of 5×10^{-6} amperes per square centimeter.

(b) After two 30-minute heating cycles at 200° and 300° C. Note relative change in peak height.

Figure 4. - Relative growth of derivative of aluminum Auger peaks with heating.

because lower beam energies better reflect surface effects and because the equipment is limited such that the maximum electron energy observable is 1500 electron volts.

RESULTS AND DISCUSSION

Interpretation of Results

A typical Auger trace for a copper - 10-percent-aluminum alloy showing the growth of the aluminum peak with temperature is shown in figure 4. References 15 and 16 justify the use of dN/dE and $N(E)$ peaks, respectively, for determining the concentration of a substance in the vicinity of metal surface.

In order to analyze these data, the ratio of the aluminum peak-to-peak height (following ref. 15) to the peak-to-peak height of the 60.2-volt copper peak was used. The data were treated in this fashion because it removes any fluctuations resulting from changes in operating conditions from one measurement to the next by normalizing to the height of the copper peak.

The effect of temperature on aluminum surface concentration is shown in figure 5.

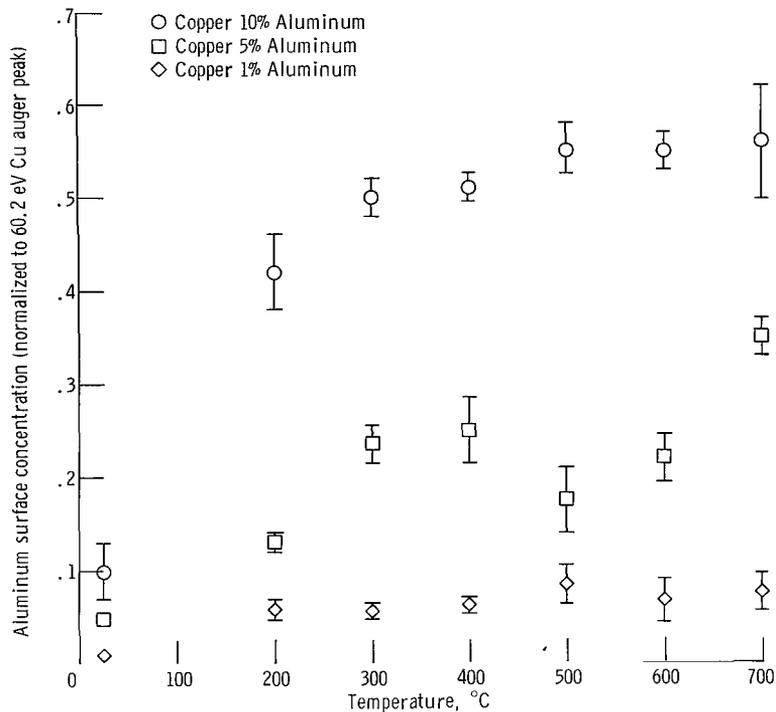


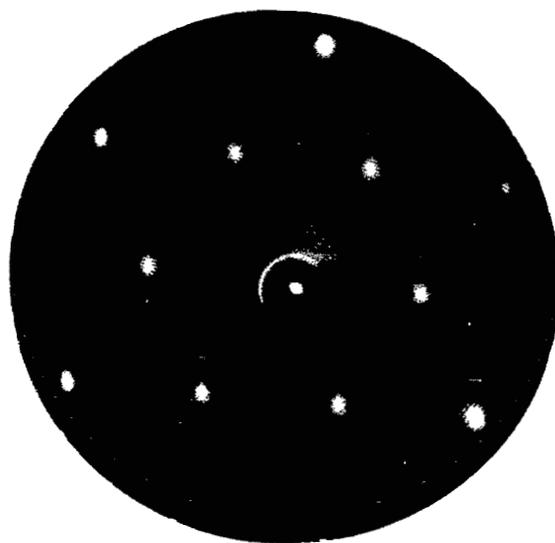
Figure 5. - Increase of aluminum surface concentration.

The error bars represent a standard deviation for repetition of the same measurement. The data are presented in terms of concentration as a function of temperature since the assumption is made that, following sputtering, the aluminum enriched region is removed and the true concentration of the alloy is observed. This gives a means for calibrating the enriched concentration. The postsputtering, normalized, aluminum peak-to-peak heights were defined to be the bulk concentration. All concentrations are calibrated by the postsputtering peak for the copper - 10-percent-aluminum alloy. The copper - 5-percent-aluminum alloy agreed to within 4 percent with this value. The postsputtering aluminum peak was unobservable for the copper - 1-percent-aluminum alloy. On this basis the data in figure 5 indicate an increase of aluminum concentration to five times the bulk value. Buckley (ref. 1) observed a similar increase in aluminum concentration in adhesion studies performed on copper-aluminum alloys where sputtering was not used to clean the surfaces.

A further justification of this interpretation for these results is shown in figure 6, which shows LEED patterns for the 5-percent and 10-percent concentration alloys following sputtering and heating to 600° C. These patterns represent a one-third monolayer aluminum coverage on the surface. (A possible representation in the direct lattice showing one-third monolayer coverage is shown in fig. 7.) In the LEED patterns ob-



(a) Copper - 5-percent aluminum alloy; beam energy, 100 volts.



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(b) Copper - 10-percent aluminum alloy; beam energy, 114 volts.

Figure 6. - LEED patterns for copper-aluminum alloys after sputtering then heating to 600° C.

served following sputtering, no additional spots were observed within the outer ring shown in figure 6. The lower intensity half-order spots observed for the 5-percent aluminum concentration pattern could indicate a partially formed layer (ref. 19). The 1-percent aluminum concentration alloy showed no additional spots in the LEED patterns. Repeating the heating procedure used in the Auger analysis and observing a LEED pattern after each heating period showed an increase of intensity of the fractional-order spots. This increase indicates an increase in concentration and substantiates the Auger observations.

Is Bulk Composition Obtained After Sputtering?

A question may arise regarding the validity of the assumption that bulk composition is obtained following sputtering. Kaminski (ref. 17) states that changes in surface concentration may occur in binary alloys resulting from differences in yields. Yield is defined as the ratio of the number of particles removed to the number of incident particles. Whener (ref. 18) points out, however, that compositional changes only occur in initial stages of sputtering and that after sputtering for long times bulk composition is reflected.

Depth of Enhanced Aluminum Concentration

As a further substantiation of these results and in an attempt to estimate the depth of

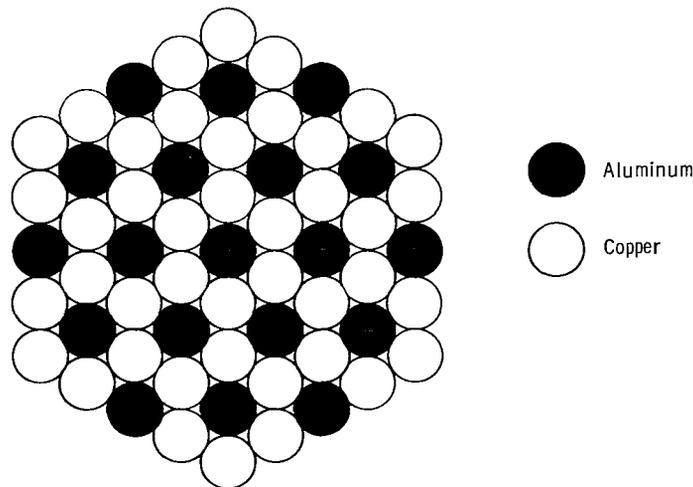


Figure 7. - Possible direct lattice structure for observed LEED pattern $R30^\circ$ (2×2) structure showing one-third monolayer coverage.

different concentrations, the escape range of the copper and aluminum Auger electrons is compared with the depth of material removed by sputtering. If the escape range of electrons were much greater than the depth of sputtered material removed, the assumption that postsputtering represented the bulk concentration could not be justified. The escape range for 72-electron-volt electrons in silver is determined in reference 20. It was found to be approximately one or two atomic layers. Copper should give similar results. The depth of material removed by 15 hours of sputtering at a current density of 5×10^{-6} amperes per square centimeter and a beam energy of 400 electron volts is estimated from yield data given in reference 21. The depth removed was estimated to be 1350 layers. Therefore, the electron escape depth is smaller than the number of layers of sputtered material removed. This indicates that the region of enriched concentration may be as low as one layer but less than 1350 layers deep. A more accurate estimation of depth of altered surface concentration should be possible by performing controlled sputtering experiments along with AES measurement of aluminum peak height change.

Change in Energy of First Plasmon Peak

Another, but not nearly as quantitative, evidence of surface segregation is a change in location of the first plasmon peak of copper with change in concentration. The first plasmon peak represents the minimum energy for collective oscillations of the electron gas (ref. 7). As such, it depends on the number of density of free electrons in a solid, which should change with composition. Table II shows the shift in position of the 19-volt copper plasmon peak after a sputtering and a heating sequence. The peak shift indicates a compositional change. Powell (ref. 8) and Klemperer and Shepherd (ref. 9) studied changes in characteristic loss spectra of copper-aluminum alloys. The results presented herein are in qualitative agreement with those of Klemperer and Shepherd. However, the comparison should not be taken too seriously, since Klemperer and Shepherd's

TABLE II. - LOCATIONS OF COPPER FIRST PLASMON
PEAK FOR EACH ALLOY

Material	Following sputtering	Following heating (600° C)
Pure copper	19.8	19.8
Cu 1 % Al	18.5	17.7
Cu 5 % Al	18.8	18.3
Cu 10% Al	19.6	17.3

samples were polycrystalline alloys and the samples in the present study are single-crystal solid solutions. In addition, because of the width of the peaks and the complexity of the spectra in these studies, absolute values of peak location cannot be considered to be accurate. In addition, copper departs from free-electron behavior (ref. 22). Alloys of materials with free-electron behavior could possibly give useful information concerning free-electron densities, Hume-Rothery rules, ref. 26), and composition.

Estimation of Activation Energy for Diffusion

A very rough estimate of the activation energy for diffusion can be made by observing the temperature at which the diffusion rate becomes appreciable (see fig. 5). This temperature is approximately 200^o C. Assuming an exponential dependence for the diffusion coefficient gives a value of approximately 0.04 electron volt for the activation energy. This value is lower than typical bulk activation energies (ref. 23) for diffusion.

Possible Mechanisms for Surface Segregation

Since the advent of LEED and AES, there is ample evidence of diffusion of interstitial contaminants to surfaces; for example, carbon has been shown to diffuse to surfaces of refractory metals (ref. 19 and 24). To date, however, little quantitative work has been done with diffusion to the surface of one component in substitutional solid solutions.

The work most directly related to the present study is segregation at grain boundaries (ref. 4), which has been studied for many years. A grain boundary bears some resemblance to a free surface in that it represents a region of discontinuity from normal crystal periodicity. The most general approach to explaining the mechanism of surface segregation is to argue that the alloying materials adjust themselves in such a manner that the free energy is minimized in the surface region (refs. 3 to 6). Although rigorous, this approach is probably less fruitful than more phenomenological explanations because the effects of various solutes on surface tension are not well known (ref. 3). Size effects and valence effects in alloys have proved to be more readily amenable to analysis. Size is important since a solute differing in size from the solvent atoms can cause stresses that can be relieved by diffusion to a surface or to a grain boundary. Valence effects can be important in that the electronic structure of a solid is altered in the region of a surface or a grain boundary; consequently, a high-valence metal atom may be attracted into a region of increased electron density by a simple coulomb interaction.

McClellan (ref. 3) has done a statistical thermodynamic analysis of solute concentration at grain boundaries with size differences as the driving force for segregation. He

finds that the equilibrium solute concentration in the grain boundary decreases with temperature but increases again as the sample cools. The present results could be interpreted to agree with McClean's analysis. In this study an artificial surface concentration is created by sputtering. As the temperature is raised, diffusion becomes important and a redistribution of atoms is possible, causing an increased surface concentration. As the temperature is raised further, a reduced equilibrium surface concentration is obtained. If the sample was rapidly quenched from this temperature, the solute concentration for the higher temperature would be observed. If it is assumed that the cooling time for high and low temperatures is the same, it would be expected that the concentration would first rise and then decrease or level off, as a function of heating temperature. This is the general behavior in figure 5. McClean's definition of concentration refers only to occupancy in the total number of stressed areas in the grain boundary region. This could explain the observed differences in maximum surface coverage with aluminum bulk concentration shown in figure 5, in that aluminum may only be able to diffuse easily in areas where there is aluminum already present and distorting the lattice. McClean considers the effects of a grain boundary to extend two or three atom layers. This may also be the case with a free surface. This question can be settled in the future by combining controlled surface sputtering with Auger spectroscopy.

It is possible to explain the observed results from purely thermodynamic considerations based on minimization of free energy. For example, Gibbs adsorption (ref. 5) could explain the observed behavior. For Gibbs adsorption, which is similar to standard adsorption, the rate of concentration increase drops with increasing concentration (ref. 4); that is, the sticking coefficient drops with coverage.

In addition, Williams and Hayfield (ref. 5) point out that conditions near a free surface differ from those in the bulk, in that it is possible for vacancy concentrations to be much higher in the region of a free surface than in the bulk. In addition the region past the surface can be considered to be a huge vacancy. If this is the case, then analyses used for bulk diffusion, which are outlined by Lazarus (ref. 23), may be applicable to surface diffusion. In this case, size and valence effects would be important driving mechanisms for vacancy diffusion. Smith (ref. 25) using a jellium model has shown that the electronic charge distribution in the region of a surface is altered in such a manner that positive ions would be attracted to the surface.

CONCLUDING REMARKS

Grain boundary and surface segregation are frequently observed. The problem with previous studies was difficulty in obtaining quantitative results. The advantage in the present study is that results are quantitative and surface structure changes can be ob-

served. AES and LEED provide the means by which future, well-controlled experiments can be performed on clean surfaces. It is hoped that, in future studies, some new quantitative information can be obtained concerning surface segregation. A program for future studies would be to select crystal systems in which size, valence, and grain orientation effects could be observed directly. The present study represents only one data point in the explanation of segregation mechanisms, since aluminum is both larger and has a higher valence than copper. However, the results observed (i. e., higher surface concentration) were expected on the basis of both size and valence considerations.

SUMMARY OF RESULTS

Segregation of aluminum in the region of the surface has been observed in the copper-aluminum alloys studied. For all samples, surface concentrations approximately five times bulk concentrations were observed. Surface concentration increased with temperature. An approximate activation energy of 0.04 electron volts was estimated. Both LEED and characteristic losses gave confirming evidence of composition changes. The direction of surface segregation followed expected trends.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, August 12, 1970,
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