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High-mass heterogeneous cluster formation by ion bombardment of the ternary alloy $\text{Au}_7\text{Cu}_5\text{Al}_4$

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The ternary alloy $\text{Au}_7\text{Cu}_5\text{Al}_4$ was irradiated with 0.1–10 keV Ar^+ and the surface composition analyzed using laser sputter neutral mass spectrometry. Ejected clusters containing up to seven atoms, with masses up to 2000 amu, were observed. By monitoring the signals from sputtered clusters, the surface composition of the alloy was seen to change with 100 eV Ar^+ dose, reaching equilibrium after 10 nm of the surface was eroded, in agreement with TRIDYN simulation and indicating that the changes were due to preferential sputtering of Al and Cu. Ejected gold containing clusters were found to increase markedly in intensity while aluminum containing clusters decreased in intensity as a result of Ar sputtering. Such an effect was most pronounced for low energy (<1 keV) Ar^+ sputtering and was consistent with TRIDYN simulations of the depth profiling. The component sputter yields from the ternary alloy were consistent with previous binary alloy measurements but showed greater Cu surface concentrations than expected from TRIDYN simulations. © 2016 American Vacuum Society. [<http://dx.doi.org/10.1116/1.4941140>]

I. INTRODUCTION

Ion sputtering of alloys and compounds changes the surface and near surface elemental compositions, leading to changes in the intensities of ejected atoms and clusters. Much work has been done early on, especially for binary noble metal alloys^{1–4} in order to identify the basic processes giving rise to the above changes. The most complete modelling of these processes was then done by Lam and coworkers^{5,6} who identified both the athermal processes (preferential sputtering, dynamic mixing, and Gibbsian segregation) and thermal processes (thermal diffusion, radiation enhanced diffusion, and radiation induced segregation) giving rise to surface and near surface compositional changes.

Many fewer experiments have been carried out on ternary alloys. Galdikas^{7,8} found that 2–5 keV Ar^+ bombardment of PdAgAu alloys caused surface concentrations to vary with ion energy and Betz and coworkers⁹ showed for a range of AgAuPd alloy compositions that including a third alloying element changes the sputtering process found for binary alloys quantitatively but not qualitatively. This idea was also demonstrated by Dudonis,¹⁰ who demonstrated the similarity between equilibrium component sputter yields from the individual elements and from binary and ternary alloys. The early experimental measurements, including the above work, were made separately by Auger electron spectroscopy or by ion scattering spectroscopy. These techniques probe different depths into the surface but together can build a good picture of the surface and near surface composition.

An alternative technique, which probes ejected neutral atoms and clusters, is sputter neutral mass spectrometry

(SNMS). Typically, an inert gas ion beam is used to sputter neutral particles which are ionized [by a nonresonant laser beam in laser SNMS,¹¹ originally called surface analysis laser ionization but now known as laser sputter neutral mass spectrometry (LSNMS)] and then measured in a time-of-flight mass spectrometer. LSNMS has four advantages for studying ion beam effects in binary and ternary alloys—the first is that much of the previous work uses Ar^+ beams to probe the surface, as typically does LSNMS, so the outcome of the present experiments can be directly compared to previous works. The second is that LSNMS suffers less from the matrix effects present in competing techniques such as SIMS. The third is that sputtered clusters carry information about the local surface environment since most dimer and trimer clusters come from neighbor or near neighbor surface atoms.¹² Finally, LSNMS has a high dynamic range so is suited to measuring cluster yields that may vary over many orders of magnitude.^{13–16}

We have previously used LSNMS, in concert with a laser ablation surface technique, LIMS, to measure distributions of clusters ejected from $\text{Au}_7\text{Cu}_5\text{Al}_4$.¹⁷ In this work, we will extend the measurements to consider the change with sputtered depth in the flux of atoms and clusters ejected from the ternary alloy by a range of ion impact energies.

II. EXPERIMENT

Measurements of the neutral sputtered flux were made on an instrument, SARISA, described previously.¹⁸ Two ion beams were used in the experiment. A low energy (100 eV–4.9 keV) Ar^+ beam from a VG EX05 ion gun was normally incident on the sample in order to damage the surface. The low impact energies were achieved by decelerating

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the ions when biasing the target with an appropriate voltage. The gun produced an ion current of 170 nA into a $350 \mu\text{m}$ beam spot, which was rastered over an area of $0.6 \times 0.7 \text{ mm}^2$ to produce a flat bottomed crater.

A second ion beam was directed into the center of the above irradiated crater in order to measure the damage. This 10 keV Ar^+ beam from an Atomika WF421 ion gun at a 60° incidence was focused to a $90 \mu\text{m}$ spot that could be raster scanned over a $0.4 \times 0.4 \text{ mm}^2$ area, so that uniform sampling of the material was ensured. The primary ion beam, with a typical current of $0.4\text{--}1.2 \mu\text{A}$ depending on the experiment, was pulsed on for 300 ns for each of the 10 000 ion beam pulses required to collect one spectrum, so that 0.03 nm of surface was removed per analysis, assuming a sputter yield of the ternary alloy of 12.¹⁹ The two ion beams were aligned *in situ* using an optical Schwarzschild-type microscope²⁰ and the alignment checked *ex situ* using optical white light interferometry.²¹

In order to measure the ejected neutrals, after the ion beam was switched off, desorbed secondary ions were swept out of the plume with a high voltage SIMS suppression pulse. Approximately 3500 ns following the end of the suppression pulse, a GAM 100EXF laser was fired and its beam intercepted the plume so that neutral sputtered atoms and clusters were photoionized and extracted into a TOF mass spectrometer. Two laser wavelengths were used in the experiment, 0.8 mJ/pulse at 157 nm when the GAM laser used F_2 gas (to obtain the energy dependence in Figs. 4 and 6) and 12 mJ/pulse at 193 nm when the laser used ArF gas (used to obtain the dose dependence in Fig. 2). Previous measurements^{14–16} have established that for many Au_nAl_m and Au_nCu_m clusters, these pulse energies were insufficient to saturate the photoionization, even for the 193 nm wavelength, but as the results of the present work are only dependent on the relative signals from photoionized neutrals the change in laser wavelength or lack of saturation during photoionization did not affect the results. The results were however dependent on the laser intensities remaining constant throughout the experiment. This condition was ensured by continual checking of the laser intensity through the course of the measurements.

During the experiments, the chamber vacuum was 10^{-8} mbar, almost all Ar from the low energy ion gun. The oxygen partial pressure, almost all from H_2O , was 5×10^{-10} mbar. The sensitivity of the peaks in the LSNMS spectra to oxidation from the vacuum system was checked. The only neutral oxide peaks observed, after extended sputter cleaning of the sample, were from AlO and Al_2O desorption. These peaks doubled in size when exposed to the vacuum for 1 min, but there were no changes in any other peaks. Given that LSNMS analysis was performed immediately after low energy Ar^+ irradiation, surface contamination from the vacuum is unlikely to affect the main results obtained in this work.

III. MODELING

TRIDYNFZR,²² a program which simulates the dynamic change of composition of a multicomponent target during

high-dose ion implantation, was used to model the ion beam induced changes in the ternary alloy. The program was first checked to ensure that changes in input parameters, such as the surface binding energy, did not unduly affect the simulated depth profiles. The sputter yield is theoretically proportional to the inverse of the surface binding energy (SBE), which is, in general, composition dependent. In TRIDYNFZR, the surface binding energy of surface atom i in a surface containing k atomic species is given by

$$\text{SBE}_i = \sum_{j=1}^k \text{SBV}_{ij} c_j, \quad (1)$$

where SBV_{ij} is the contribution of atom j to the surface binding energy of atom i and c_j is the surface atomic fraction of atom j . The elemental surface binding energies, ΔH_i^s , used in the simulation came from Kudriavtsev²³ (Al 3.19 eV, Cu 3.48 eV, and Au 4.13 eV) with the values of SBV_{ij} chosen to be the means of the individual elemental binding energies [i.e., with $\Delta H^f = 0$ in Eq. (2)]. Simulations were also run with the values of SBV_{ij} given by

$$\text{SBV}_{ij} = \frac{1}{2} (\Delta H_i^s + \Delta H_j^s) + \Delta H^f, \quad (2)$$

where ΔH^f are the heats of formation of equiatomic binary alloys of elements i and j . For AuCu, AuAl, and AlCu, the values of ΔH^f are 0.76,²⁴ 0.37,²⁵ and 0.21 eV/atom,²⁶ respectively. When these values are used in Eq. (2), the calculated surface concentrations after sputtering to equilibrium only change by a few percent from the values found when ΔH^f was set to zero.

IV. RESULTS AND DISCUSSION

Figure 1 shows the LSNMS spectrum from a clean $\text{Au}_7\text{Cu}_5\text{Al}_4$ target sputtered with $10^{13} \text{ ions cm}^{-2}$ 10 keV Ar^+ at 60° incidence and the neutrals postionized with 193 nm light. The major peaks below 200 amu are due to Al, Al_2 , Cu, CuAl, CuAl_2 , Cu_2 , Cu_2Al , Cu_3 , and Au. Above 200 amu, the most prominent peaks are AuAl and Au_2Al followed by

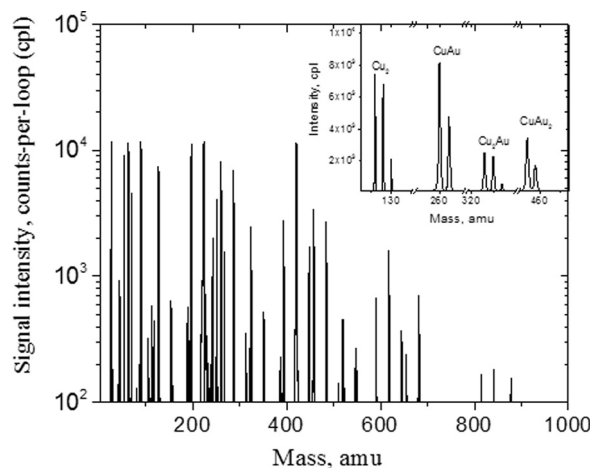


FIG. 1. Laser SNMS spectrum of clean $\text{Au}_7\text{Cu}_5\text{Al}_4$ sample. Insert demonstrates the behavior of Cu and Cu_2 containing clusters (see Sec. IV).

AuCu, AuCuAl, Au₂Cu, and Au₂CuAl. Lesser peaks above 200 amu originate from both mixed clusters AuAl₂, AuCu₂, AuCu₂Al, Au₂Cu₂, Au₃Al, Au₃CuAl, and elemental clusters Au₃ and Au₄. The count rates from the atomic species Al, Cu, and Au are saturated under the conditions used to collect Fig. 1. So, the decrease in 3 orders of magnitude in signal intensity from the atomic species to 8-mers such as Au₆CuAl (1272 and 1274 amu) is an underestimate of the real decrease.

There are both similarities and differences between the spectrum shown in Fig. 1 and spectra obtained previously for sputtered binary alloys. First, as discussed above, cluster signals decrease rapidly with increasing cluster size. We have previously verified that the sputter yield, Y_n , of a cluster containing n atoms, can be approximated by

$$Y_n \sim n^{-\delta}, \quad (3)$$

where δ ranges from 3.4 and 4 to 7 for Au_n clusters ejected from pure gold, Au₄Al, and Cu_xAu_{1-x} alloys, respectively.¹⁴⁻¹⁶ Equation (3) is only strictly true for sputtered elements; so, to take account of sputtering from alloys, the simplest solution is to assume a random association of desorbing atoms. In that case, the yield, $Y(x,y,z)$, of a Au_xCu_yAl_z cluster from Au₇Cu₅Al₄ would be¹⁷

$$Y(x,y,z) \sim \frac{n!}{x!y!z!} \left(\frac{7}{16}\right)^x \left(\frac{5}{16}\right)^y \left(\frac{4}{16}\right)^z (x+y+z)^{-\delta}. \quad (4)$$

The decrease in the cluster intensities over more than 3 orders of magnitude can be understood by the power law decrease in the overall cluster intensity made even more dramatic due to the low probability of ejecting dilute constituents, such as Al in the ternary alloy.

To illustrate a difference between sputtering the ternary alloy and previous measurements on binary alloys, we have previously shown that for Ar⁺ irradiated Au_xCu_(1-x) alloys¹⁴ the sputtered clusters are more copper rich than the bulk concentration. If we consider, for example, the intensities of Au_nCu_m dimers, trimers, and tetramers ejected from Au_{0.6}Cu_{0.4}, which has approximately the same relative fractions of Au and Cu as the ternary alloy if the Al and contribution is neglected, then we would expect higher signals from Cu₂ and Cu₂Au compared to CuAu and CuAu₂, respectively, for example. Higher signals are not seen for the more Cu rich clusters in the spectrum presented in Fig. 1. This difference between cluster ejection from the binary and ternary alloy may well come from the influence of Al in the ternary alloy. We have shown¹⁶ that Au_mAl_n clusters sputtered from Au₄Al are gold rich compared to the expected 80% Au concentration. This arises because of the increased stability of AuAl clusters compared to pure Au clusters and the enrichment of Au in the sputtered surface. Since Al containing clusters are also more stable than Cu containing clusters, e.g., Au₂Cu and AuCu have binding energies of 3.45 and 2.7 eV,²⁷ less than those of Au₂Al and AuAl, 5.8 and 3.81 eV,²⁷ respectively, any competition between ejection of Al and Cu

containing clusters would be expected to favor the Al containing clusters leading to a decrease in intensity of ejected Cu rich clusters.

Figures 2(a) and 2(b) show the change in intensity of sputtered Au rich, and Cu and Al rich clusters as a function of sputtering time for 100 eV Ar⁺ irradiation. The Au rich clusters are seen to increase with sputtering time, the Al rich clusters decrease with sputter time, and copper rich clusters show an initial increase, peaking at about 20 s sputtering followed by a decrease over longer sputtering times. Gades and Urbassek¹² related homonuclear dimer sputter yields to the clustering or agglomeration probability, p . Later²⁸ this concept was extended to heteronuclear dimers through the use of sputter correlation coefficients, χ , so that the heteronuclear dimer yield, Y_{ij} , is given in terms of atomic sputter yields, Y_i and Y_j , of atoms i and j , by

$$Y_{ij} = p_{ij}\chi_{ij}Y_iY_j. \quad (5)$$

For 1 keV Ar⁺ sputtering of Au, Cu, Au₃Cu, and Cu₃Au, they found that p_{ij} was 0.03–0.04 relatively independent of

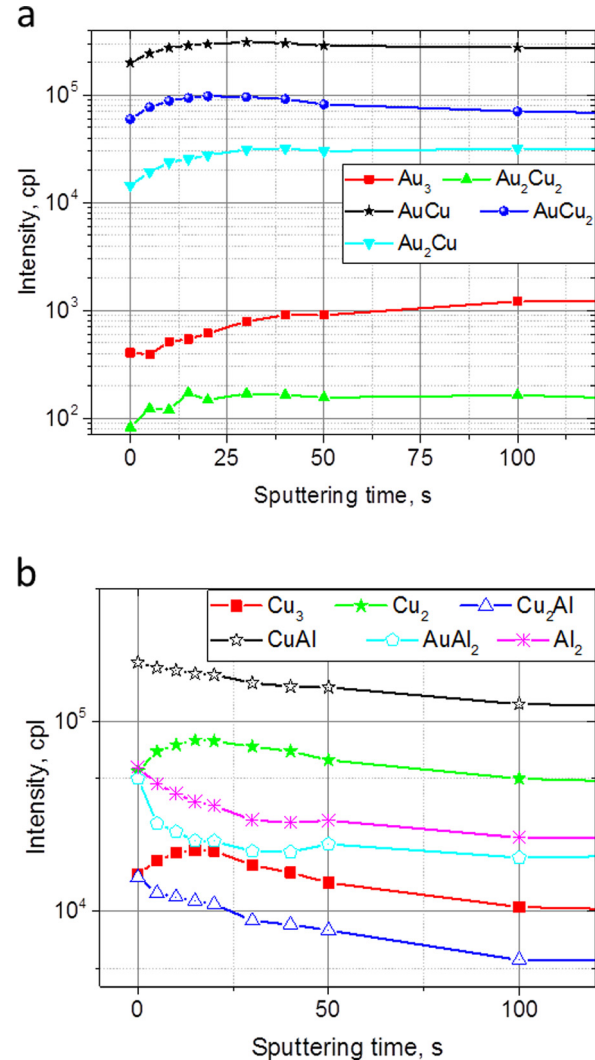


Fig. 2. (Color online) Variation of the LS-NMS signal from (a) gold rich clusters and (b) copper and aluminum rich clusters sputtered from an Au₇Cu₅Al₄ sample by 100 eV Ar⁺.

target composition while χ_{ij} was almost constant for heteronuclear cluster, AuCu, for all target compositions. If we extend this concept to the clusters shown in Fig. 2, we may expect, for example, that the AuCu₂ cluster arises from an AuCu cluster coming together with a Cu atom during the sputter event. The trimer is bound more strongly than the dimer and atom so its formation is possible. The AuCu₂ signal variation with sputter time would then be the product of the AuCu and Cu variations since both p_{AuCu_2} and χ_{AuCu_2} would be expected to be constant over the surface composition change shown in Fig. 2. This concept is illustrated in Fig. 3 for a large number of the clusters measured. In Figs. 3(a)–3(c), the ratio of the signals from the nominated clusters are plotted as a function of 100 eV Ar⁺ sputter time. The data in Fig. 3(a) show, for example, that the variation of AuCu₂ divided by the variation of AuCu shows a similar variation with dose as the Cu atomic signal does. In general, Fig. 3 shows the variation of the signals with sputter time from clusters Au_nCu_mAl_n, Au_iCu_jAl_k, and Au_{n-k}Cu_{m-j}Al_{n-k} are related by

$$\frac{\text{Au}_n\text{Cu}_m\text{Al}_n}{\text{Au}_i\text{Cu}_j\text{Al}_k} \sim \text{Au}_{n-i}\text{Cu}_{m-j}\text{Al}_{n-k}. \quad (6)$$

This could be interpreted as the coming together of Au_iCu_jAl_k and Au_{n-k}Cu_{m-j}Al_{n-k} during sputter ejection, although the dimer and trimer signals may also come from the breakup of larger ejected clusters. Equation (6) is not a universal rule since the AuCuAl trimer does have the same dose dependence as that of CuAl multiplied by Au, or AuAl multiplied by Cu, but does not have the same dose dependence as AuCu multiplied by Al. It is then tempting to postulate that, in general, the AuAlCu trimer comes from the concerted emission of the AuCu dimer and Al atom. As a further example, the dose dependence of AuCu₂ is consistent with AuCu coming together with Cu during desorption, not Cu₂ coming together with Au.

The results of the TRIDYNFZR dose dependence simulation are shown in Fig. 4 for 100 eV Ar⁺ irradiation of the ternary alloy. The sputter yield of Au is seen to increase by a factor of 3 whereas the Cu and Al signals decrease by factors of 30% and 50%, respectively, with the change in sputter yield is essentially complete after sputtering $8 \times 10^{16} \text{ cm}^{-2}$. This dose compares well with the dose dependence in Fig. 2. A fluence of 10^{17} cm^{-2} resulted in 3.1 nm of the target being eroded. The fact that the depth which is eroded to achieve equilibrium is not significantly greater than the depth down to which 100 eV Ar⁺ deposits energy into the alloy, approximately 2 nm, indicates that the processes underlying the change are athermal, i.e., preferential sputtering, dynamic mixing, and bombardment induced Gibbsian segregation.

From Fig. 2(a), it can be seen that the signal from the AuCu cluster increases by a factor of approximately 1.5 as the surface is sputtered to equilibrium by 100 eV Ar⁺. The change in signal from the commencement of sputtering to equilibrium is shown in Fig. 5 as a function of Ar⁺ energy, so the AuCu signal is seen from Fig. 5(b) to give a change of a factor of 1.6 for 100 eV Ar⁺ energy as

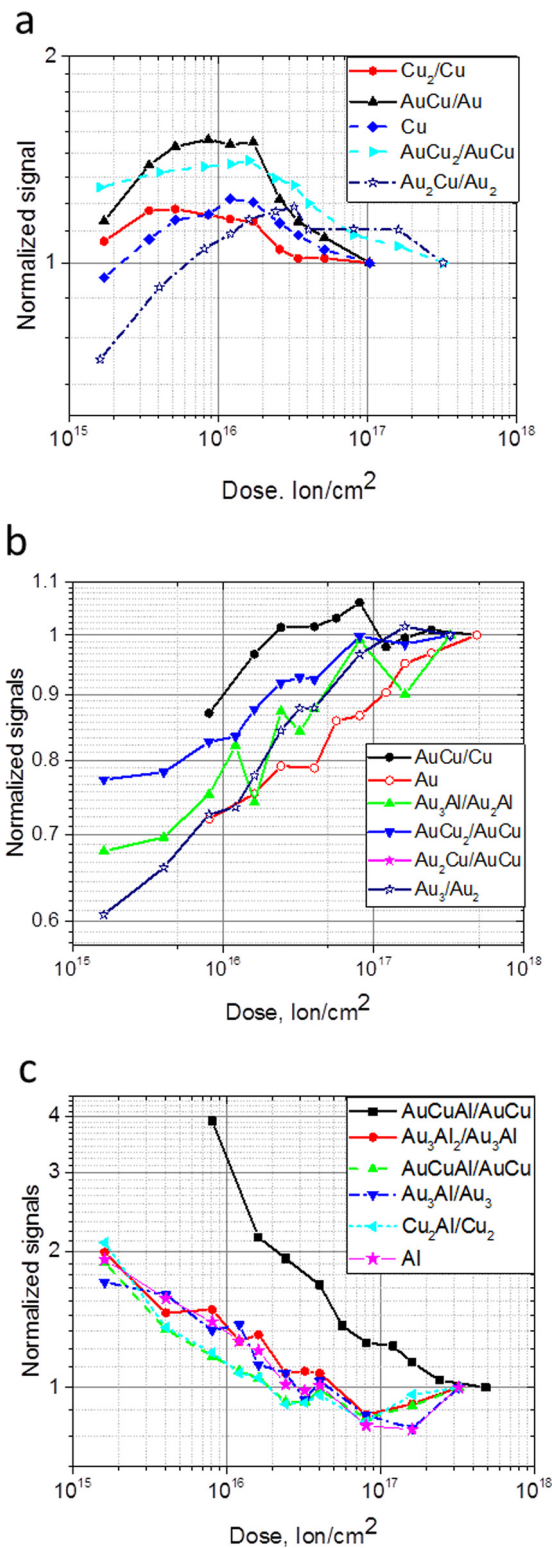


Fig. 3. (Color online) Intensity of first cluster indicated in the legend divided by intensity of second cluster and normalized to unity at large sputter times. The variation with sputter dose matches the behavior of (a) Cu, (b) Au, and (c) Al.

expected. The increase in signals from Au rich clusters with sputtering and the decrease in Al and Cu rich clusters is seen, from Fig. 5, to not only be true for 100 eV Ar⁺ sputtering but to be a general phenomenon, and to become more marked at lower sputter energies. The fact

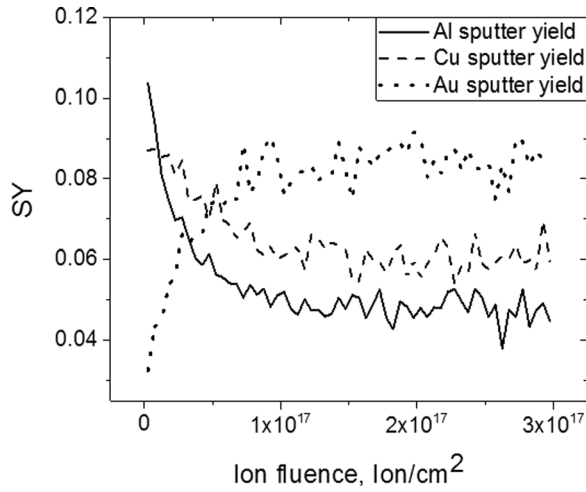


Fig. 4. TRIDYNFZR simulation of the component sputter yields as a function of ion fluence for 100 eV Ar^+ sputtering of $\text{Au}_7\text{Cu}_5\text{Al}_4$.

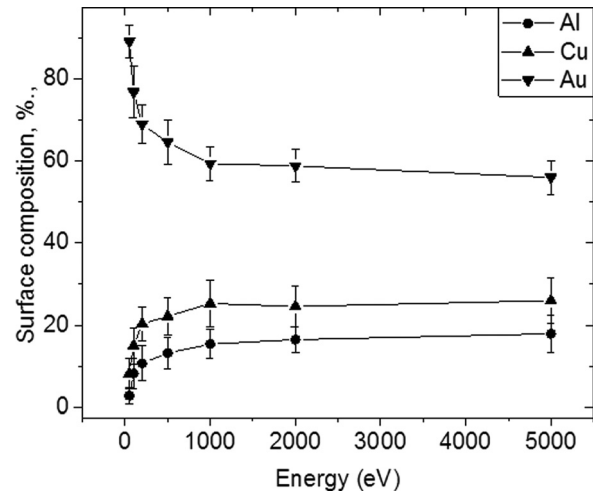


Fig. 6. TRIDYNFZR simulations of the variation in surface composition after sputtering to equilibrium at the indicated Ar^+ energies.

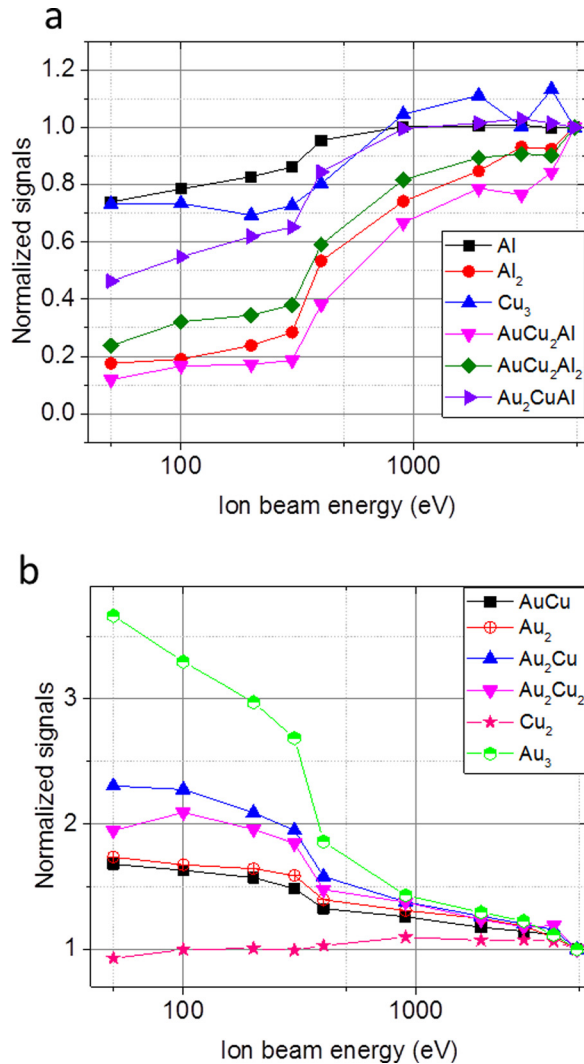


Fig. 5. (Color online) Flux of clusters after sputtering $\text{Au}_7\text{Cu}_5\text{Al}_4$ to equilibrium by Ar^+ at the indicated ion energies. The signals from (a) Al rich and (b) Au and Cu rich clusters are normalized with respect to their values for 4.9 keV Ar^+ sputtering.

that the cluster signal enhancement (or depletion) for lower ion energies matches the behavior of the individual elements confirms the behavior found in Fig. 3 and again points to a simple model for cluster ejection, in which smaller clusters aggregate with atoms during sputtering to form larger clusters.

The energy range over which the change occurs in Fig. 5 is the same as the range over which large surface concentration changes occur in TRIDYN simulations, as shown in Fig. 6, showing that the change in cluster signals is related to the surface enrichment of Au and surface depletion of Cu and Al due to preferential sputtering. Indeed, the simulations in Fig. 6 and the experimental results shown in Fig. 5 follow the same trend as found by Mousel *et al.*²⁹ who simulated the sputtering of NiW and CuW alloys by 65–1030 eV Ar^+ finding that W was enhanced in the top nanometer of both alloys and that the enhancement increases with decreasing ion energy.

The ratio of component sputter yields, Y_A and Y_B , in a multicomponent system is experimentally given by

$$\frac{Y_A}{Y_B} = \frac{c_A^{\text{bulk}} c_B^{\text{surf}}}{c_B^{\text{bulk}} c_A^{\text{surf}}}, \quad (7)$$

where c_A^{bulk} and c_A^{surf} are the bulk and surface concentrations of element A. Theoretically, the component sputter yield ratio is given by

$$\frac{Y_A}{Y_B} = \left(\frac{M_B}{M_A} \right)^{2m} \left(\frac{U_B}{U_A} \right)^{1-2m}, \quad (8)$$

with U_A and M_A being the surface binding energy and mass of element A and m being in the range from 0.05 to 0.25. Table I shows some known sputter yield ratios for single and binary alloys in comparison with data obtained in current work for ternary compound.

Dudonis and Pranevicius¹⁰ found that the component sputter yield ratios for 2 keV Ar^+ sputtered AuAgPd alloys were similar to those found in sputtered binary alloys and

TABLE I. Sputter yield ratios for single element alloys calculated from Eq. (8), for binary alloys from data in the literature and for the Au₇Cu₅Al₄ ternary alloy, taken from simulation results in Fig. 6 or from experimental measurements using Eq. (7).

	$\frac{Y_{Au}}{Y_{Cu}}$	$\frac{Y_{Au}}{Y_{Al}}$	$\frac{Y_{Cu}}{Y_{Al}}$
Single alloy	0.71	0.56	0.78
Binary alloy (Refs. 30 and 31)	1–1.1	<1	0.67
Ternary alloy (simulation)	0.59	0.5	0.84
Ternary alloy (experiment)	0.89	0.97	0.65

sputtered individual elements. We also find similarity between the experimental results for the binary and ternary alloy but these do not agree with simulations, especially for results involving gold. This difference for sputtered CuAu alloys may arise from the competition between preferential sputtering of Cu in Cu rich alloys and preferential sputtering of Au which dominates in Au rich alloys.³² This competition arises from the preferential sputtering of the lower mass element versus the bombardment induced Gibbsian segregation and hence increased sputtering of Au, which has a lower surface energy than Cu.³²

V. SUMMARY AND CONCLUSIONS

The ternary alloy Au₇Cu₅Al₄ has been sputtered by 0.1–4.9 keV Ar⁺ and ejected atoms and clusters detected using laser sputter neutral mass spectrometry. For 100 eV Ar⁺ sputtering, the dimer and trimer cluster signals came to equilibrium after a depth of order of the ion range was eroded, in agreement with TRIDYN simulation, and their variation with sputter time was consistent with aggregation of atoms with smaller clusters during sputtering to form larger clusters. The distribution of cluster yields increased greatly for lower sputter energies, with desorption of Au rich clusters being increased and Al rich clusters being reduced, mirroring the change in surface concentration with preferential sputtering as modeled with TRIDYN. The similarity between component sputter yields for binary and ternary alloys was confirmed.

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