CONF-860522--12

Submitted to the Seventh International Conference on Plasma-Surface
Interactions in Controlled Fusion Devices, Princeton NJ, May 5-9 , 1986

HIGH PLASMA-FLUX ELEVATED TEMPERATURE
SPUTTERING OF Cu-Li ALLOYS*

CONF-860522--12
JUN 11 1986

A. R. Krauss, D. M. Gruen, M. H. Mendelsohn,
Argonne National Laboratory

CONF-860522--12

R. Conn, D. Goebel, Y. Hirooka and K. Leung
University of California at Los Angeles

DE86 012120

Copper-lithium alloys ranging in composition from 3-12 at.% Li have been exposed to sputtering by 3×10^{16} - 6×10^{17} 100 eV He+/cm²-sec at temperatures of 300-500°C at the UCLA PISCES plasma device. Weight loss and optical spectroscopy techniques were used to determine the sputtering-induced erosion of the binary alloys relative to pure copper. It was found that the weight loss of the alloy and the amount of copper in the plasma as measured by emission spectroscopy never exceeded that of pure copper and in some cases was reduced by a factor of five or more.

Post-irradiation analysis by Auger electron spectroscopy and scanning electron microscopy show a correlation between lithium surface depletion, surface roughening, weight loss, and partial erosion yields as measured by plasma emission spectroscopy. The results are explained in terms of form-

*This work supported in part by the U.S. Department of Energy,
BES-Materials Sciences, under Contract W-31-109-Eng-38.

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ation of a lithium overlayer as a result of the elevated temperature and plasma ion bombardment. The formation of the overlayer is diffusion limited and depends on the sample conditioning, sample temperature, lithium concentration, method of preparation and plasma flux. The degree to which the copper erosion is reduced depends on the Li overlayer thickness, which is expected to depend on the sputtering yield of lithium on a copper substrate, the ion fraction of the sputtered lithium, the extent of subsurface lithium depletion and the duty cycle of the plasma device.

INTRODUCTION

The use of self-sustaining low-Z coatings has been proposed [1-7] as a means of reducing sputtering-induced erosion of plasma-interactive components in a fusion device. For selected alloys such as Cu-Li and Al-Li, the minority component will segregate very strongly, forming a complete overlayer even when present in relatively dilute concentration in the bulk. It has been demonstrated both experimentally [4,8] and by computer simulations [2,3,9], that an overlayer 1-2 monolayers thick is sufficient to eliminate most of the sputtering of the underlying material. Calculations using the REDEP computer code [7,10] indicate that, unlike the low-Z materials, a limiter consisting of a self-sustaining lithium layer on a copper substrate exhibits almost no dimensional change resulting from sputtering and redeposition. For light ion impact, the same is true for tungsten. However, tungsten is subject to a runaway self-sputtering cascade if the plasma edge temperature exceeds ~50 eV. For the Cu-Li system, the threshold energy for runaway self-sputtering is 1.5-2.5 x higher than that of tungsten.

It has been shown previously that the predicted lithium layers form on Cu-Li and Al-Li alloys and that resulting reduction in the sputtering rate of the majority alloy component is in good agreement with values predicted by the TRIM computer code [4-6,9-13]. In the case of Cu-Li alloys, it has been observed in ion beam experiments that for, 100-3000 eV He^+ and Ar^+ fluxes in the range 10^{12} - 10^{14} ions cm^{-2} sec^{-1} , the copper erosion is reduced by 3-4x compared with pure copper. A large fraction of the lithium is sputtered as secondary ions which are trapped at the surface, thereby reducing the lithium loss rate. However, some of the surface lithium is lost and must be replenished by bulk diffusion processes.

The time required for the layer to form, and the temperature required to maintain it during sputtering, are highly dependent on the mass, energy and flux of the incident ions. The time required to deplete the bulk lithium inventory sufficiently to inhibit overlayer formation depends on the erosion rate, the lithium secondary ion fraction, and the depletion depth. At a given temperature, the depletion depth is given as [14] $d=D/v$ where v is the surface recession velocity and D is the effective diffusion coefficient. Writing the latter quantity in terms of the bulk diffusion coefficient, $D=D_0 \exp \{-E_d/kT\}$, and using the value $D_0=0.766 \text{ cm}^2\text{s}^{-1}$ and $E_d = 1.16 \text{ eV}$ for Cu-Li [11], it is found that for a surface recession velocity of, e.g., 1 A/sec, lithium must be depleted to a depth of 6 mm at 450°C or 26 cm at 600°C before the surface lithium concentration will decrease to a value determined by the preferential partial sputtering yields. It should be noted that the actual depletion depth may be considerably greater as a result of radiation-enhanced diffusion and grain boundary segregation [14] and that as a result of the protective effect

of the overlayer and the trapping of secondary ions, the erosion rate is expected to be lower than that determined by the physical sputtering yields of the components of the binary alloy. For beta aluminum-lithium (48-52 at.% Li), the diffusion coefficient is 4-5 orders of magnitude greater than for Cu-Li [15] and the 1 A/sec recession velocity corresponds to a depletion depth of 100-1000 cm at 415°C. The detailed effects of diffusion, segregation and preferential sputtering in these materials are discussed elsewhere [12,16].

Since plasma fluxes at tokamak limiters (5×10^{16} - 1×10^{18} particles $\text{cm}^{-2} \text{sec}^{-1}$) are much higher than the particle flux available from conventional ion guns, and because behavior in a plasma is not the same as behavior under ion beam irradiation, it is necessary to determine the formation, protection, and depletion characteristics of the lithium layer in a plasma producing a light ion particle flux comparable to that of a tokamak fusion reactor. Recently, a plasma device, PISCES [17,18] has been developed which continuously produces plasma with values of density (10^{11} - $2 \times 10^{13} \text{ cm}^{-3}$) and electron temperature (5-20 eV) characteristic of the edge plasma in modern fusion experiments. In this paper, we report first results of Cu-Li exposure to such a continuous, high flux plasma. Discussion of sample mounting [21], flux measurement techniques [17,18], and additional plasma spectroscopy results [22] are discussed elsewhere.

EXPERIMENTAL

The high particle flux was provided by the PISCES plasma irradiation device at UCLA. The samples were prepared by two different methods. In the first series, up to 25 grams of Cu-Li alloy were prepared at a time by arc melting a high lithium concentration (~ 70 at. % Li) master alloy with

additional copper. The samples were turned and remelted a total of four times each to insure uniformity. Typically, less than ??% of the lithium was lost in this process. In order to produce larger samples for the second series of experiments, a procedure was developed [19,20] for melting up to 250 grams at a time in an electric furnace. Since the cooling time was longer in this process, slightly more (~ ??%) of the lithium was lost, although it was possible to prepare samples containing up to 19 at. % Li. It has been pointed out [20] that this second procedure may produce materials with substantially more free lithium than the arc-melting process.

The sample is inserted into a helium plasma and initially allowed to be electrically floating. Consequently, the sample is very near the plasma potential and no sputtering occurs. ***Secondary electron emission*** results in heating to the temperature desired for the particular run. When the desired temperature is reached, the sample is biased -100 V, resulting in sputtering by the impact of 100 eV He^+ . Fluxes ranged from $\sim 4 \times 10^{16}$ to 6×10^{17} ions $\text{cm}^{-2} \text{sec}^{-1}$. The temperature is maintained, typically within $\pm 5^\circ\text{C}$ by forced air cooling. After a period ranging from 0.5-4 hours, the sample is removed and weighed. During the second series of experiments, the 3520 Å neutral copper emission line and the 6708 Å neutral lithium emission line were monitored spectroscopically, using an optical multi-channel analyzer. In order to prevent metal deposition on the window, a shutter was placed behind the viewport and only opened for a few seconds at a time to take spectral measurements.

RESULTS

The results of the weight loss measurements are summarized in Table I for 100 eV He^+ incident on pure Cu and Cu-Li alloys. Except as denoted by an asterisk, the samples were exposed to the stated fluence in one continuous irradiation. For the Series 1 alloys denoted (*), the values reported were at the end of the second one-hour exposure to the plasma. For the Series 2 alloys, (*) denotes the weight loss during the last of a series of one hour exposures to the plasma.

For the pure copper samples, the weight loss was nearly constant, regardless of the incident flux, fluence, or sample temperature. For the Cu-Li alloys, at relatively low temperature (350°C), the lithium diffusion is too slow to allow replenishment within the monolayer removal time. Consequently, the sputtering behavior is nearly identical with that of pure copper. For the 3 at.% Li alloy, raising the temperature from 350°C to 400°C at relatively low flux, resulted in a reduction in weight loss.

As the flux was raised to 2×10^{17} ions/ cm^2 -sec, the 3 at.% Li alloy was no longer able to supply lithium at an adequate rate and the weight loss was no lower than that of pure copper. However, if the temperature was 400°C or greater, the weight loss for the 6 and 12 at.% Li alloy was up to 3.8x smaller than for pure copper, in accord with previous calculations using the TRIM computer code [2,3,9] and with earlier experimental results at lower temperatures and much lower fluxes [4,8]. At a flux of 6×10^{17} He^+ / cm^2 -sec, a temperature of 450°C was required to maintain the protective effect. It should be noted, however, that this flux is close to the maximum value anticipated for a conventional tokamak limiter, and that the sputtering rate for the He^+ plasma used is about 5x higher than that of D^+ at the same impact energy.

During the initial heating, the 12% Series 2 alloys but not the arc-melted Series 1 alloys gave off a red glow corresponding to the 6708 Å emission line of lithium. In order to determine if a significant fraction of the observed weight loss for the 12% samples was due to lithium evaporation, one of the 12% Series 2 samples was heated to 450°C by the plasma without bias for 1/2 hour. Within the experimental accuracy of the balance used, negligible weight loss (0.4 mg) was measured, although droplets of lithium were observed to form on the surface. It has been recently determined [23] that the vapor pressure of lithium in the Cu-Li alloy is approximately 6 orders of magnitude lower than that of elemental Li.

The Series 2 samples were exposed to a number of consecutive irradiations and removed just long enough to record the weight loss and recycle the vacuum system. The pure copper and the 12% Li sample irradiated at 400°C (Sample A) showed essentially the same weight loss, independent of the fluence. The measured erosion rate for pure copper, Cu-6 at.% Li (Sample B) and Cu-12 at.% Li (Sample C) at 450°C during irradiation by 6×10^{17} 100 eV $\text{He}^+ \text{cm}^{-2} \text{sec}^{-1}$ are shown in Ref. 22. The Cu-6 at.% Li sample B initially had the same weight loss as pure copper, declining eventually to about half that of copper. The 12% sample C irradiated at 450°C initially showed a weight loss greater than that of pure copper, declining to about 2/3 that of pure copper at a fluence of 1.08×10^{21} ions/cm².

The intensity of the 3520 Å copper emission line was measured and it was found that with a plasma flux of $6 \times 10^{17} \text{cm}^{-2} \text{sec}^{-1}$ at 400°C, there is no reduction in the influx of Cu into the plasma for the 12% lithium alloy. At 450°C the situation is quite different. There was a large amount of neutral lithium seen in the plasma when sputtering first began

and following each time the sample was removed for weighing as shown in Ref. 22. However, although the initial rate of lithium loss appears to have been large, no copper at all could be seen in the plasma for nearly the first hour of irradiation, corresponding to a reduction in the copper erosion rate of at least 4x. Bohdanský and Roth have reported a reduction in the copper erosion rate by up to 50x for 1 keV D⁺ bombardment of Cu-16 at.% Li [23].

As the lithium layer thinned, copper emission lines could be seen, although the intensity decreased with time. For the Cu-6 at.% Li Sample B at 450°C, (Fig. 1) the intensity of the copper emission line decreased steadily with time until it had dropped to about 1/5 of the initial value. The intensity of the neutral Li emission line also decreased steadily, indicating a possible increase in the secondary ion fraction of the sputtered lithium, in accord with earlier experiments [7].

The flux was monitored both by means of a Langmuir probe and by continuously measuring the total sample current. Since the measured sample current includes the secondary electron emission, the ratio of the measured current densities is:

$$J_{LP}/J_S = 1 + \gamma \quad (1)$$

where J_{LP} is the Langmuir probe current density, J_S is the measured sample current density, and γ is the secondary electron yield. Since the majority of the secondary electrons originate within 15-30 atomic layers of the surface, and lithium has one of the lowest secondary electron yields of any of the elements, a macroscopically thick lithium coating results in a material with a very low secondary electron yield. If the lithium coat-

ing, however, is only a few monolayers thick, the reduced work function associated with an alkali metal coating results in a very high secondary electron yield. The low work function corresponds to a condition of maximum surface charge transfer, leading to a maximum in the secondary ion fraction of the sputtered lithium [24]. The sample current measurement may therefore be used as a diagnostic for the existence and thickness of a lithium overlayer. For the 12% Li sample at 450°C (Sample C), the initial secondary electron yield was very low, in accord with the observed formation of lithium droplets and the high lithium loss rate. As the amount of copper observed in the plasma decreased with increasing dose, the secondary electron yield continuously increased, reaching a value greater than 0.9. For the Cu-6 at.%Li Sample B of Fig. 1, the secondary electron yield increased throughout the run as the Cu emission line decreased. The secondary electron yield again exceeded 0.9 by the end of the run. Similar results have been obtained in glow discharge experiments.

Post-irradiation Auger analysis and electron microscopy were used to measure morphological and compositional changes associated with the plasma exposure. It was found that samples with lowest weight loss had the highest degree of surface roughness. It has been reported [25] that surface roughening associated with redeposition results in reduced erosion rates of copper. However, substantial surface roughening was observed in pure copper samples which did not exhibit reduced weight loss. It is therefore to be concluded that regardless of the mechanism by which the lithium acts to reduce the erosion of copper, there is a very significant difference in the sputtering-induced erosion of pure copper and Cu-Li alloys.

Electron micrographs of the three lithium-bearing samples designated A, B, and C are shown in Fig. 2. For Sample A, the Cu-12% Li alloy irradiated at 400°C, the weight loss and Cu plasma emission were almost as high as that of pure copper, and the surface was overall very smooth with some roughening occurring only along the grain boundaries. For Sample B, the 6% Li sample irradiated at 450°C, the weight loss was 53% that of pure Cu and the copper concentration in the plasma was approximately 5x lower than for pure Cu. The surface consists largely of a roughened texture with a small amount of smooth area. For Sample C, the 12% Li sample irradiated at 450°C, the surface is very rough and the initial copper erosion was so low that it could not be measured spectroscopically, although a large amount of lithium was seen in the plasma. In previous ion beam experiments [4-8] in which it was possible to use Auger spectroscopy to monitor the surface lithium concentration during sputtering, it was found that reduced Cu^+ emission was directly related to the presence of lithium at the surface during sputtering. It is to be expected therefore that the samples which exhibited the greatest degree of protection will be depleted in surface lithium upon subsequent room temperature analysis. Auger spectra of samples A, B, and C are shown in Fig. 3 and it is seen that this expectation is realized: The surface of Sample A is nearly pure lithium with almost no copper in the first atomic layer. Samples B and C exhibit progressively greater degrees of lithium depletion.

Scanning Auger microscopy (Fig. 4) of these samples shows that the surface of Sample A is largely covered with a thick lithium layer which completely obscures the 61 eV Auger line of copper over most of the sample surface. Sample B shows patches of high Li concentration, although most

of the surface area is largely copper. Only a small amount of Li is seen on the surface of sample C.

DISCUSSION

We have observed two conditions in which the presence of a lithium layer reduces the copper erosion: The formation of an initially very thick lithium layer was sufficient to reduce the influx of copper into the plasma to the point that we were unable to detect it spectroscopically. The rapid loss of lithium in this regime requires a very high diffusion rate and large inventory of available lithium for sustained operation. One possible means of doing this, consisting of a porous tungsten block with liquid Li coolant channels, has been suggested previously [26].

After extended operation, or for alloys with a lower bulk lithium content, a conditioning process was repeatedly observed to occur. During this process, the secondary electron yield increased and both the copper and lithium observed in the plasma decreased steadily with time. The final value of the secondary electron yield was too high to account for by any means other than the formation of a reasonably uniform lithium overlayer 1-2 monolayers thick. This thickness is in accord with the requirements for the production of an alkali metal coating in which the alkali metal sputters largely as secondary ions which are therefore trapped at the surface [27,28]. The calculated reduction in copper erosion for a 1-2 monolayer lithium coating [2-3,9-13] is in good agreement with the observed reduction in copper emission from the plasma. Erosion and redeposition calculations [7,10] using the REDEP code indicate that the observed reduction in copper erosion corresponds to a threshold plasma edge temperature for runaway self-sputtering 1.5-4x that of tungsten.

Lithium depletion depends on the secondary ion fraction of sputtered lithium and on the depth of the altered layer. For high temperature operation, this depth may exceed the thickness of the plasma-interactive component, and the depletion lifetime is determined only by the time to deplete the entire bulk lithium inventory.

Lithium replenishment appears to occur via bulk and grain boundary diffusion. At high fluxes at a given temperature, bulk diffusion may be too slow to provide adequate replenishment and grain boundary segregation may then be the dominant replenishment mechanism. In that case, it is necessary for the lithium to spread across the grain surface in order to provide protection to the substrate material. This process requires high grain boundary and surface diffusion rates and small grain size. Composites of lithium-bearing binary alloys and refractory metals such as tungsten have been suggested [26] as a means of providing adequate strength at the temperatures required to maintain the desired diffusion rate and as a means of inhibiting the grain coarsening which occurs in copper and most copper alloys as a result of prolonged heating [26]. Other composite materials such as W-Al-Li have intrinsic lithium diffusion rates several orders of magnitude higher than that of lithium in copper. As an additional benefit TRIM calculations indicate that for some of the composites under investigation, the threshold for runaway self-sputtering corresponds to plasma-edge temperatures as high as 500 eV. The self-sputtering and redeposition properties of such materials are described in detail elsewhere [26,29].

SUMMARY

It has been demonstrated that, under the appropriate conditions, there exists a protective mechanism for Cu-Li alloys which results in a reduction of sputtering-induced erosion of the copper component. The observed reduction in copper loss corresponds to light ion sputtering and redeposition properties comparable to those of a low-Z material, and a calculated threshold for runaway self sputtering significantly better than that of tungsten. For high Li concentration alloys, the protection begins as soon as the sample reaches an elevated temperature, and the rate of copper erosion is reduced by a factor of five or more. For lower Li concentration alloys, there is a conditioning period during which the rate of copper erosion drops steadily until it is at least 4x lower than that of pure copper. The length of the conditioning period and the required operating temperature depend on the sample temperature, incident flux and, based on past experiments, the mass and energy of the incident particles. The protective effect has been observed at particle fluxes comparable to, and with erosion rates in excess of, those expected at the limiters of large tokamaks such as TFTR and JET.

The experimental results are in accord with the predicted formation of a self-sustaining lithium overlayer. The formation rate of this overlayer is determined by the relative rates of lithium diffusion to the surface and sputtering-induced desorption from the surface. Consequently, the process is expected to depend strongly on sample temperature as well as a number of other parameters relating to method of preparation, bulk lithium inventory, duty cycle of the plasma, etc. The parameter space in which the protective effect occurs needs to be more completely determined and, if necessary, enlarged by the use of modified materials which provide

higher lithium diffusion rates, smaller grain size, higher lithium secondary ion fraction and better high-temperature strength.

TABLE I

		Li Conc. (At. %) (°C)	Temp. (Ions/cm ² -sec)	Flux (Ions/cm ² -sec)	Fluence (Ions/cm ²)	Weight Loss (mg-cm ² /ion)
SERIES 1						
	0	350	4x10 ¹⁶	5.76x10 ²⁰	1.23x10 ⁻²⁰	
	0	400	4x10 ¹⁶	5.76x10 ²⁰	1.52x10 ⁻²⁰	
	0	400	2x10 ¹⁷	7.2x10 ²⁰	1.71x10 ⁻²⁰	
	0	450	2x10 ¹⁷	7.2x10 ²⁰	1.62x10 ⁻²⁰	
	3	350	4x10 ¹⁶	5.76x10 ²⁰	1.20x10 ⁻²⁰	
	3	400	4x10 ¹⁶	5.76x10 ²⁰	6.94x10 ⁻²¹	
	3	400	2x10 ¹⁷	7.2x10 ²⁰	1.89x10 ⁻²⁰	
	3	450	2x10 ¹⁷	7.2x10 ²⁰	1.89x10 ⁻²⁰	
	6	350	4x10 ¹⁶	5.76x10 ²⁰	9.5x10 ⁻²¹	
	6	400	4x10 ¹⁶	5.76x10 ²⁰	4.5x10 ⁻²¹	
	6	400	2x10 ¹⁷	7.2x10 ²⁰	4.5x10 ⁻²¹	*
	6	450	2x10 ¹⁷	7.2x10 ²⁰	6.7x10 ⁻²¹	*
	12	450	2x10 ¹⁷	7.2x10 ²⁰	5.8x10 ⁻²¹	*
SERIES 2						
	0	350	6x10 ¹⁷	2.16x10 ²¹	1.24x10 ⁻²⁰	
	0	400	6x10 ¹⁷	2.16x10 ²¹	1.04x10 ⁻²⁰	
	0	450	6x10 ¹⁷	2.16x10 ²¹	1.08x10 ⁻²⁰	
	0	450	6x10 ¹⁷	2.16x10 ²¹	1.23x10 ⁻²⁰	
B	6	450	6x10 ¹⁷	2.16x10 ²¹	5.8x10 ⁻²¹	*
	12	350	6x10 ¹⁷	2.16x10 ²¹	1.0x10 ⁻²⁰	
A	12	400	6x10 ¹⁷	2.16x10 ²¹	1.14x10 ⁻²⁰	*
C	12	450	6x10 ¹⁷	2.16x10 ²¹	7.6x10 ⁻²¹	*

*Last exposure of a series

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FIGURE CAPTIONS

- Fig. 1 - Intensity of the 3520 Å copper emission line vs dose for Cu-6 at.% Li at 450°C.
- Fig. 2 - Scanning electron micrographs of samples A, B, and C taken after exposure to the PISCES plasma.
- Fig. 3 - Room temperature Auger spectra of samples A, B, and C taken after exposure to the PISCES plasma.
- Fig. 4 - Scanning Auger maps of the lithium and 920 eV copper Auger lines (samples A and B) and 61 eV copper line (sample C).