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Temperature and Composition Dependence of the High Flux Plasma Sputtering Yield of Cu-Li Binary Alloys

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Because of its very high thermal conductivity, actively cooled copper is an attractive plasma-interacrtive material for long pulse fusion devices such as ETR and devices with very high wall power loadings, such as reversed-field pinches (RFPs) and the proposed compact ignition torus (CIT). Pure copper however, has an unacceptably low threshold energy for runaway self-sputtering. Low Z materials such as graphite and beryllium are not subject to runaway self-sputtering, but suffer from high light ion erosion rates and very non-uniform redeposition. It has been suggested that strongly segregating alloys such as Cu-Li might be used to provide a low-Z self-sustaining coating while maintaining the desirable redeposition, thermal and mechanical properties of the majority alloy component.

High flux deuterium plasma sputtering and ion beam experiments have been performed on Cu-Li alloys to determine if the reduction in copper erosion previously predicted and observed in low flux ion beam experiments occurs at particle fluxes representative of an RFP first wall or tokamak limiter. Partial sputtering yields of the copper and lithium components have been measured as a function of alloy composition and sample temperature using optical plasma emission spectroscopy, weight loss and catcher foil techniques. It is found that the lithium sputtering yield increases with increasing sample temperature while the copper yield decreases by as much as two orders of magnitude. The temperature required to obtain the reduction in copper erosion is found to be a function of bulk lithium concentration. Consequences of these experimental results for anticipated erosion/redeposition properties are calculated, and the Cu-Li alloy is found to compare favorably with conventional low-Z materials.

MASTER

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INTRODUCTION

For plasma edge temperatures in excess of 50 eV there are very few materials which may be used for plasma-interactive components. Tungsten undergoes runaway self-sputtering for edge temperatures in excess of 50 eV and stainless steel can not be used at edge temperatures over 10 eV [1]. Additionally, it is vitally important to prevent contamination of the plasma with high Z impurities if net energy gain is to be achieved. Low Z materials such as graphite and beryllium limit plasma energy losses due to line radiation and are not subject to runaway self-sputtering, but they are subject to other problems:

Graphite, because of its chemical affinity for hydrogen and because of its porosity, acts as an enormous sink for hydrogen, making it difficult to control the plasma density and in some cases trapping the fill gas (e.g. deuterium) while filling the vessel with hydrogen (protium) trapped during manufacturing and handling. This problem can be partially alleviated through the use of "diamond-like" carbonized layers [2], but these coatings are only 1000-3000 A thick, and it is difficult to imagine how such a coating could survive in a reactor environment or even in some of the next-generation long pulse machines. Both graphite and beryllium are subject to extensive erosion by light ion sputtering. Because of their relatively long ionization mean free paths, they migrate extensively through the plasma and do not redeposit uniformly. It is estimated for example, [1] that for a plasma edge temperature of 100 eV, the leading edge of an INTOR limiter made of beryllium would be subject to gross erosion (not including redeposition) in excess 160 cm/year. Assuming that the redeposited material has properties similar to that of the virgin material, there would be a net erosion of about 30 cm/year as shown in Fig. 1.

Similar results are obtained for graphite if chemical erosion effects are ignored. Because of its relatively poor thermal conductivity, a graphite limiter operates at a very high temperature (e.g. ~2400 C for CIT) even under normal conditions. Much more extreme temperatures are encountered during a disruption. Graphite is an excellent high temperature material and might be usable at high temperature if it retains its as-fabricated properties. It has however been noted [3] that redeposited graphite behaves more like soot than the original material. Such material, if it is structurally stable, is likely to be an excellent trap for hydrogen, complicating the density control problem, although it will probably be thermally ablated.

Beryllium is not subject to the chemical and mechanical problems of graphite, but represents potential problems in terms of toxicity and melt layer stability. It is therefore desirable to explore alternatives to these two materials for use in the next generation of fusion devices.

Actively cooled copper structures are capable of withstanding very high thermal loads. Copper represents a neutron activation problem, but this may not be more severe than the beryllium toxicity for near-term devices and can be handled by much the same methods A major difficulty however, for the use of copper as a plasma-interactive material is its very low threshold for runaway self-sputtering.

It has been demonstrated by means of low flux (10^{12}-10^{14} cm^{-2} sec^{-1}) ion beam experiments [4-10] that under the appropriate conditions, lithium segregation in a copper-lithium alloy is capable of producing a self-sustaining lithium overlayer which significantly reduces the copper erosion. When properly conditioned, the lithium sputters primarily as secondary ions which are trapped at the surface by the sheath potential and magnetic fields tangential to the surface. The conditioning process occurs as a result of prolonged high temperature sputtering. Once a surface has been conditioned, the high lithium secondary ion fraction and reduction in Cu sputtering appears to persist at lower temperatures than those required for conditioning. Because of the short ionization mean free path for copper, it is calculated that sputtered copper is redeposited very uniformly, and it is projected that Cu-Li alloys may be usable to plasma edge temperatures of 200 eV [10,11] even though elemental copper has a high self-sputtering yield.

It is additionally projected that Cu-Li has a lower light ion sputtering yield and more uniform redeposition than graphite or beryllium, better density control than graphite, and as an actively cooled material, better thermal response than beryllium. Although the thermal and mechanical properties have not been systematically investigated, the material has machining properties comparable to those of iron. The question of attachment to an actively cooled substrate also seems to be much simpler than that of graphite since it is fairly easy to produce a diffusion bond between Cu-Li alloys and pure copper.

It is the purpose of the present experiments to determine if the reduction of substrate sputtering, high lithium secondary ion fraction and consequent low erosion rate, and conditioning phenomena previously observed in low flux ion beam experiments on Cu-Li alloys persist at the

high particle fluxes charactersitic of large magnetic confinement devices. It was also desired to determine the effect of the lithium concentration, sample temperature and particle flux on the time required for the conditioning process, and to determine if the values used in the REDEP calculation for Cu-Li shown in Fig. 2 are substantiated for light ion fluxes in the range 10^{16} - 10^{18} cm^{-2} sec^{-1}.

EXPERIMENTAL

The PISCES device at UCLA was used to provide a plasma flux ranging from 4×10^{16} to 6×10^{17} ions cm^{-2} sec^{-1} of 100 eV He$^+$. The relative level of copper and lithium in the plasma was determined spectroscopically. Additionally, the sample was removed at periodic intervals from the vacuum chamber for weight loss measurements. The spectroscopic analysis was not available during the initial lower flux (4×10^{16} and 2×10^{17} ions cm^{-2} sec^{-1}) series of experiments. Preliminary spectroscopic results and weight loss measurements at the higher fluxes were reported earlier [3,12], and the parameters of the PISCES device are described elsewhere [13,14]. The plasma was maintained at a low density and low electron temperature, resulting in a copper mean free path of approximately 25 cm. This condition guarantees that negligible redeposition of the sputtered material occurs [19] and that the observed erosion is a measure of the sputtering behavior. The Cu-Li alloy samples were prepared [15] by casting, using a Cu$_4$Li master alloy and pure copper to produce material containing approximately 3, 6, and 12 at.% Li, as determined by atomic absorption analysis. An optical multi-channel analyzer (OMA) and grating spectrometer were used to monitor the intensity of the Cu (320 nm) and Li (670.8 nm) emission lines. A shutter was placed over the window and opened only during the measurement in order to eliminate the possibility of transmission loss due to contamination of the window. Additionally, the He (667.8 nm) line was monitored for reference purposes.

A second series of experiments was performed at the Max Planck Institut fur Plasmaphysik in Garching, West Germany, using 100 and 1000 eV D$^+$ ion beams. Sputtered material was collected on a silicon collector plate. The amount of sputtered copper was determined by Rutherford backscattering (RBS) analysis, and the lithium was analyzed by nuclear reaction analysis (NRA).Preliminary results of the Garching experiments were reported previously [16].

RESULTS

At the lower He$^+$ fluxes (4x10^{16} and 2x10^{17} ions cm^{-2} sec^{-1}), the PISCES weight loss measurements for Cu-Li showed reduced copper sputtering relative to pure copper at 400 C and to some extent, even at 350 C. With a flux of 6.25x10^{17} He ions cm^{-2} sec^{-1}, the intensity of copper line emission observed spectroscopically in the PISCES plasma for the nominal 6 at.% and 12 at.% Li samples was found to be almost identical with that of the pure copper reference sample at 400 C. At 450 C however, the samples behaved quite differently.

For a 6 at.% Li sample (Fig. 4), the copper sputtering yield was initially close to that of pure copper. With continued sputtering however, the level of copper in the plasma decreased continuously, and when the measurement was terminated, had dropped by ~6x relative to the value for pure copper and ~8x relative to the value at the start of the measurement. Weight loss measurements were made at intervals ranging from 1 to 4 hours. The weight loss decreased with time in direct proportion to the intensity of the copper emission line; Within the experimental scatter the measured weight loss for each sampling period was proportional to the average copper emission intensity during that period. The level of copper in the plasma for the pure copper sample was dose-independent. The sample current density was monitored by means of a Langmuir probe which was periodically inserted in front of the sample. The measured current density was very nearly constant, indicating stable operation of the plasma discharge. The intensity of the 670.8 nm Li emission line was also monitored. The intensity of the Li line emission dropped continuously, roughly paralleling the Cu line intensity.

It has been observed in previous SIMS/AES experiments [10] that continued sputtering of Cu-Li results in an increase in the Li secondary ion fraction by at least a factor of 4x relative to the initial value. Interpreting the data of Fig. 4b as the result of increasing lithium secondary ion fraction, it is estimated that the Li secondary ion fraction exceeded 80% at a dose of 8x10^{21} ions cm^{-2}. Consequently it is found that for high flux plasma sputtering, the copper erosion is reduced by an amount in accord with the previous experiments [4-10] and with the input values used for the calculation shown in Fig. 2.

For a sample containing 12 at.% Li sputtered at 450 C, initially the level of copper emission was below the detection limit of the spectrometer.

There was an initially high level of line radiation corresponding to neutral lithium, dropping rapidly with time. After prolonged sputtering, copper appeared in the plasma, rising briefly to the level observed for pure copper samples and then dropping with time. During this experiment, the sample was removed once an hour for weight loss measurements, as indicated by the arrows in Fig. 5. This procedure appears to disrupt the conditioning process as evidenced by increased lithium sputtering, and in some cases, increased copper sputtering after re-insertion into the plasma. However, the subsequent time dependence of the copper and lithium signals after re-insertion appears to behave in a manner qualitatively similar to that of the Cu-6 at.% Li sample of Fig. 4, i.e. both the copper and lithium emission decrease continuously.

A sample of Cu-16 at.% Li was sputtered by beams of 100 and 1000 eV D^+ at a flux of 1×10^{16} cm^{-2} sec^{-1} at MPI-Garching. The absolute copper and lithium sputtering yields were measured as a function of temperature. The lithium yield was temperature-independent and was determined to be 0.13 for 100 eV impact and 0.10 for 1000 eV. By applying a bias to the target to trap the lithium secondary ions, it was determined that the lithium appeared to be sputtered primarily as neutrals. The copper yield was ~2x lower than that of pure copper from room temperature to 450 C and then started to decrease further , reaching a value ~100x lower than that of pure copper at 550 C as shown in Fig. 6. Sputtering for a total of ~20 hours produced no observable change in the copper sputtering yield, although the accumulated dose was much less than that obtained in the PISCES experiments.

By turning off the ion beam and measuring the rate at which lithium was evaporated onto the catcher foil as a function of temperature, it was possible to determine the rate of lithium evaporation. The results are shown in Fig. 7, compared with the expected evaporation rate of elemental lithium. The Li evaporation rate for the Cu-Li alloy is much lower than that of elemental Li, corresponding to an activity coefficient of 10^{-8}-10^{-9}.

DISCUSSION

The concentration profile of the lithium which segregates to the surface is not known and furthermore is expected to be dependent on the bombardment conditions. As a simple model for the Cu-Li system, we have used a modified version [6] of the TRIM computer code to calculate the expected copper sputtering yield, assuming that the lithium first fills the outermost atomic layer as expected thermodynamically [17], and then

progressively proceeds to fill only the second layer, third layer etc. Fig. 3 shows the calculated lithium and copper sputtering yields as a function of lithium "layer thickness" for 200 eV He$^+$ and 3 keV Ar$^+$ impact. The reduction in the copper sputtering yield of 4-6x relative to that of unalloyed copper observed in previous low flux ion beam experiments [4-10] corresponds to a lithium "layer thickness" of ~1.5 monolayers. A REDEP [11] calculation was performed based on these experimental results and an assumed lithium secondary ion fraction of 90%. The results of the calculation are shown in Fig. 2.

Although the fluxes in the current experiments are 4-6 orders of magnitude higher than those used earlier [4-10], the results are qualitatively quite similar. Initially, the lithium forms an apparently thick coating which sputters primarily as neutral [10] Li atoms while significantly reducing the copper sputtering yield. As shown in Fig. 3, it is calculated that approximately three monolayers of lithium are sufficient to produce the degree of suppression in the copper sputtering yield observed in the D$^+$ experiments on Cu-16 at.% Li [16].

It is to be expected [18] that as a result of dipole-dipole repulsion, the effective lithium adsorbate charge and consequently, the secondary ion fraction of lithium in contact with the copper substrate will decrease with increasing surface lithium concentration. For multiple lithium layers the predominant lithium-lithium bonds are purely metallic, and a very low lithium secondary ion fraction is seen experimentally [18], and expected theoretically. However, it should be noted that although a multi-atomic layer Li coating is consistent with the observed copper and lithium sputtering yields and small secondary ion fraction observed for the 12% and 16% Li samples, it is inconsistent with the evaporation data of Fig.7.

Two other mechanisms are capable of reducing the copper sputtering yield: surface roughening and compound formation. Substantial post-irradiation surface roughening was observed in those Cu-Li PISCES samples which exhibited reduced copper sputtering [12]. For pure copper, it has been shown [19] that surface roughening can reduce the sputtering yield by $\leq 5x$. Compound formation alters the surface binding energy and consequently, also affects the sputtering yield. It has been calculated [20] that radiation-induced segregation in Cu-Li results in the subsurface formation of a region of increased lithium concentration. The lithium concentration in this region may exceed the value corresponding to precipitation of the compound Cu_4Li [21]. However, this mechanism has been observed to result in a reduction in the copper sputtering yield by no more than a factor of ~5x [22]. If all three mechanisms (lithium overlayer formation, surface roughening and compound formation) are invoked, it is possible to obtain 100x reduction in the copper sputtering yield for a lithium overlayer 1-1.5 monolayers thick.

If the temperature is high enough to maintain a lithium coating during prolonged sputtering, a conditioning process occurs [8,10], presumably as the result of radiation-induced segregation and cascade mixing. As a result of the conditioning, the surface is modified so that the lithium is sputtered primarily as secondary ions. Although the conditioning process is not yet understood, it has been observed that the required dose depends strongly on both the flux and sample temperature [9]. As a result of the high flux in the current experiments, high bulk diffusion rates and consequently, high temperatures are required in order to obtain any improvement over the sputtering behavior of pure copper. Clearly, it would be preferable to operate copper structural components at a temperature less than 450 C. In the low flux ion beam experiments [8], it was observed that once a conditioned surface had been produced, the temperature could be lowered by 100-200 C while retaining the protective effect observed to occur as a result of lithium surface layer formation. Additionally, it may be argued on the basis of the phase diagram that for a bulk Li concentration close to 20 at. %, compound formation is expected to occur at relatively low temperature [16]: For Cu-16 at.% Li, the solid solution phase ends at 450 C, the temperature at which the onset of the sharp decrease in sputtering yield occurred. For Cu-20 at.% Li, the solid solution phase ends at ~250 C. It may therefore be possible to reduce the required operating temperature of Cu-Li plasma-interactive components by increasing the lithium concentration.

SUMMARY

Depending on the alloy concentration, the temperature and the particle flux, the surface composition and sputtering characteristics of Cu-Li alloys can vary from essentially that of pure copper to that of pure lithium, with little or no copper sputtering. For high lithium surface concentrations, the lithium sputters largely as neutral atoms with approximately the same sputtering yield as elemental lithium. As the lithium surface concentration decreases, the lithium secondary ion fraction increases and the copper sputtering yield although no longer negligible, is much lower than that of elemental copper. It appears possible to maintain this "conditioned" surface for a long time although the conditioning process is complex and the lifetime of the conditioned surface has not been quantified.

More work needs to be done to determine the reason for the very dramatic decrease observed in the copper sputtering yield for high lithium concentration alloys, the nature of the conditioning process, and the conditions under which high Li secondary ion fractions may be obtained. Additionally, it remains to be determined how best to condition the surface *in-situ* , and whether surface conditioning can be done *ex-situ.* The lifetime of the protective surface has not been determined, although it has been demonstrated that for limiter applications it is equivalent to at least several hundred hours in current low-duty-cycle machines. For first wall applications, the equivalent dose corresponds corresponds to several years of operation. Cu-Li alloys seem ideally suited for use in Reverse-Field - Pinches which produce very high thermal loads but have only modest ($\sim 10^{16}$ cm^{-2} sec^{-1}) particle fluxes at the first wall.

It has been demonstrated that copper-lithium alloys are capable of reducing the copper sputtering yield by at least 4-6x compared with pure copper, and that lithium secondary ion fractions >80% can be obtained as a result of surface modification associated with prolonged sputtering. REDEP calculations based on these results project that for the INTOR limiter design, Cu-Li alloys may be used with plasma edge temperatures up to ~200 eV, compared with a maximum of ~50 eV for tungsten [11]. For edge temperatures 50 eV$\leq T_e \leq$ 200 eV, copper-lithium, as a result of its low light-ion erosion yield under fusion conditions and of the relatively short ionization mean free path for copper, is expected to be subject to both much less gross and net erosion than Be or graphite.

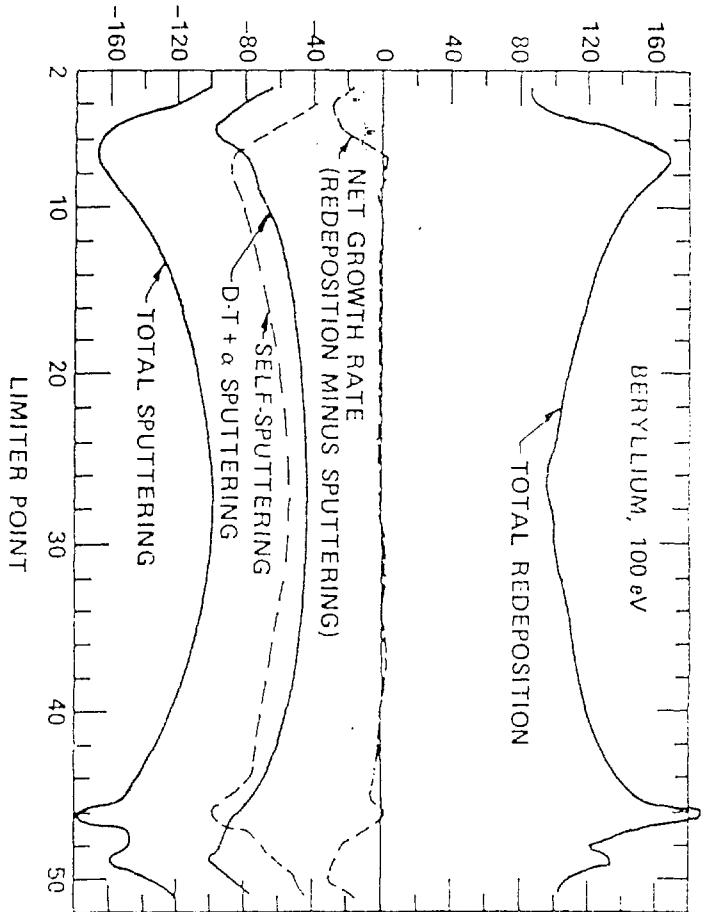# ACKNOWLEDGEMENT

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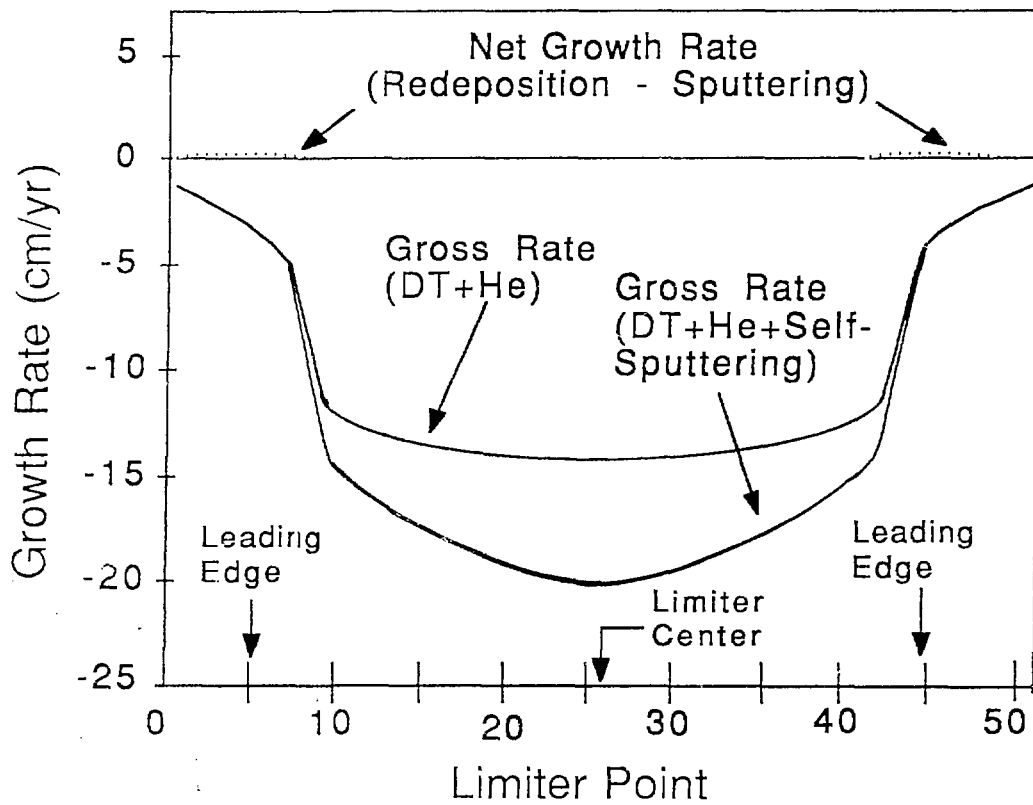
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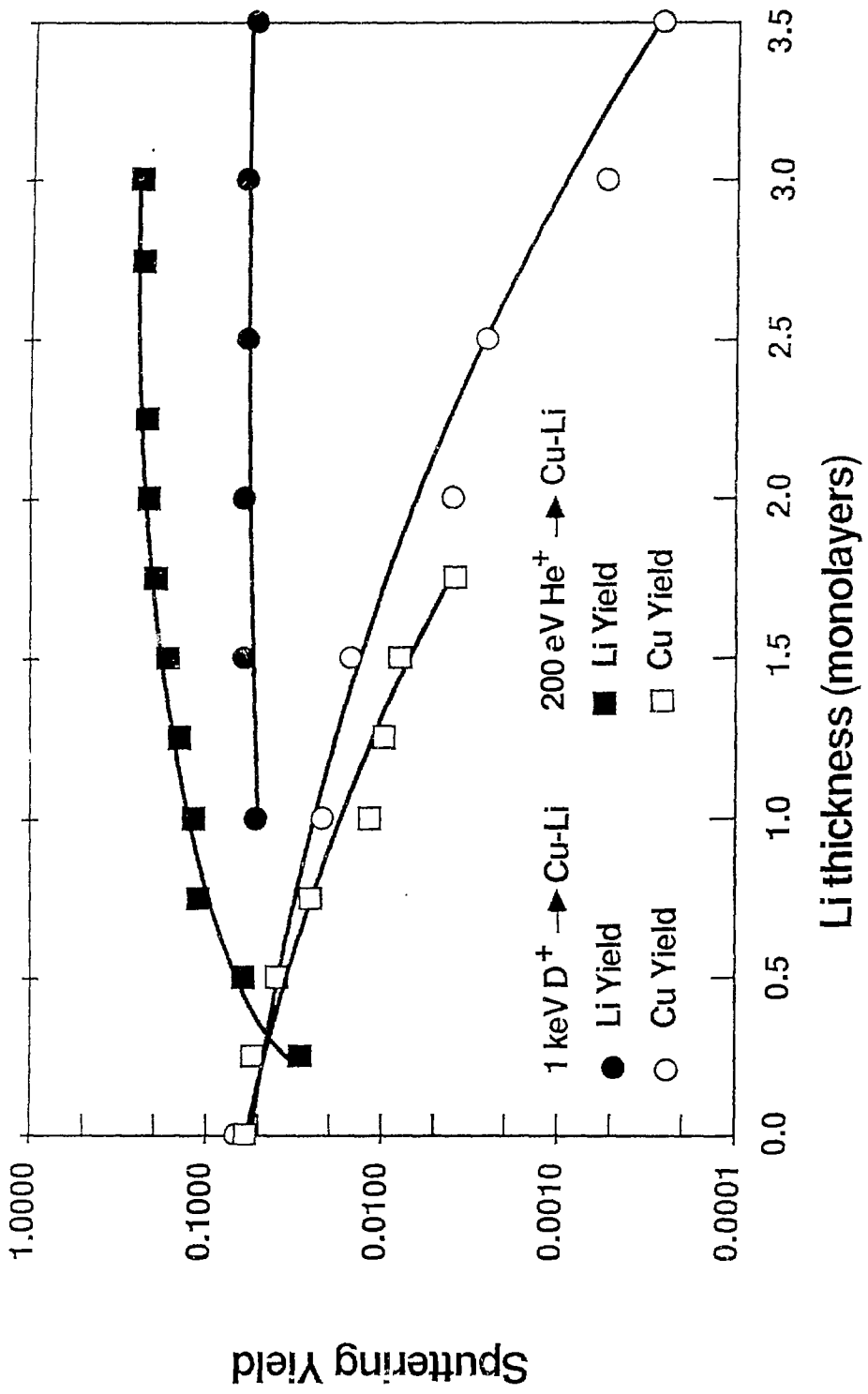
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FIGURE CAPTIONS

1. Calculated (REDEP) erosion and redeposition properties of a beryllium limiter for INTOR with a plasma edge temperature of 100 eV. (After Ref. 1)

2. Calculated (REDEP) erosion and redeposition properties of a copper-lithium limiter for INTOR with a plasma edge temperature of 100 eV. (After Ref. 11)

3. Sputtering yields of copper and lithium as calculated by TRIM as a function of lithium layer thickness for an incident beam of 1000 eV D^+ (●), and 200 eV He^+ (■).

4. (a) Intensity of copper emission lines in the PISCES plasma for pure copper (O), and Cu-6 at.% Li (■) at 450 C. (b) Intensity of Li (●) emission lines corresponding to the Cu-6at.% Li sample of (a).

5. (a) Intensity of the copper (320 nm) emission line for Cu-12 at.% Li at 450 C. (b) Intensity of the lithium (670.8 nm) emission line for the same sample.

6. Copper sputtering yield for 100 eV and 1000 eV D^+ incident as a function of sample temperature. The open figures represent pure copper and the filled figures represent Cu-16 at.% Li.

7. Evaporation rate of lithium as determined by catcher foil measurements for pure lithium and Cu-16 at.% Li.
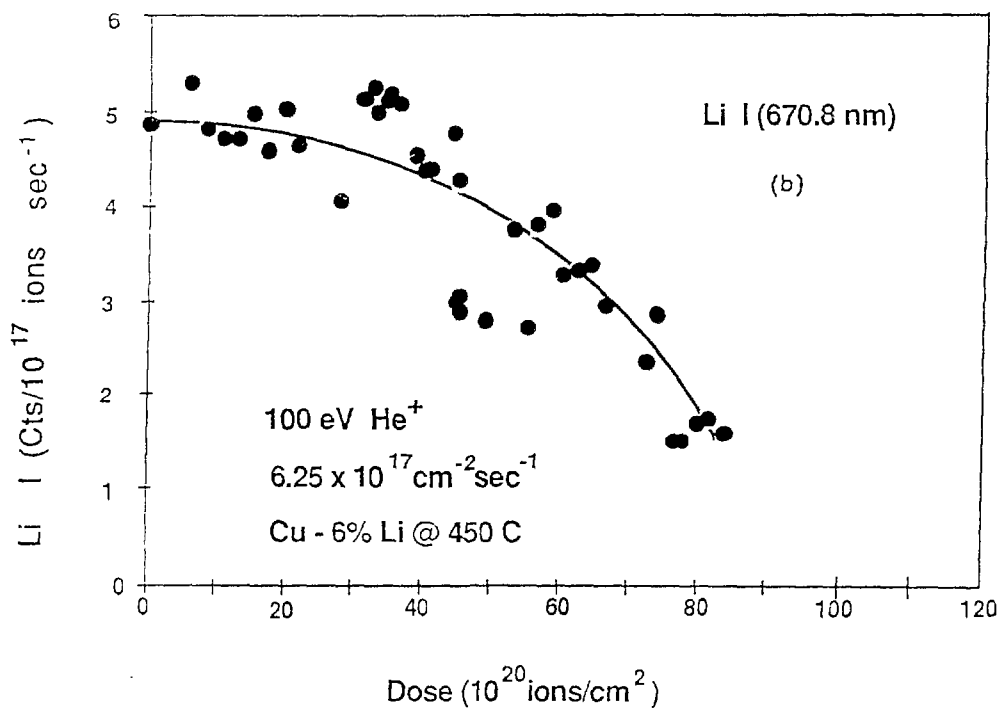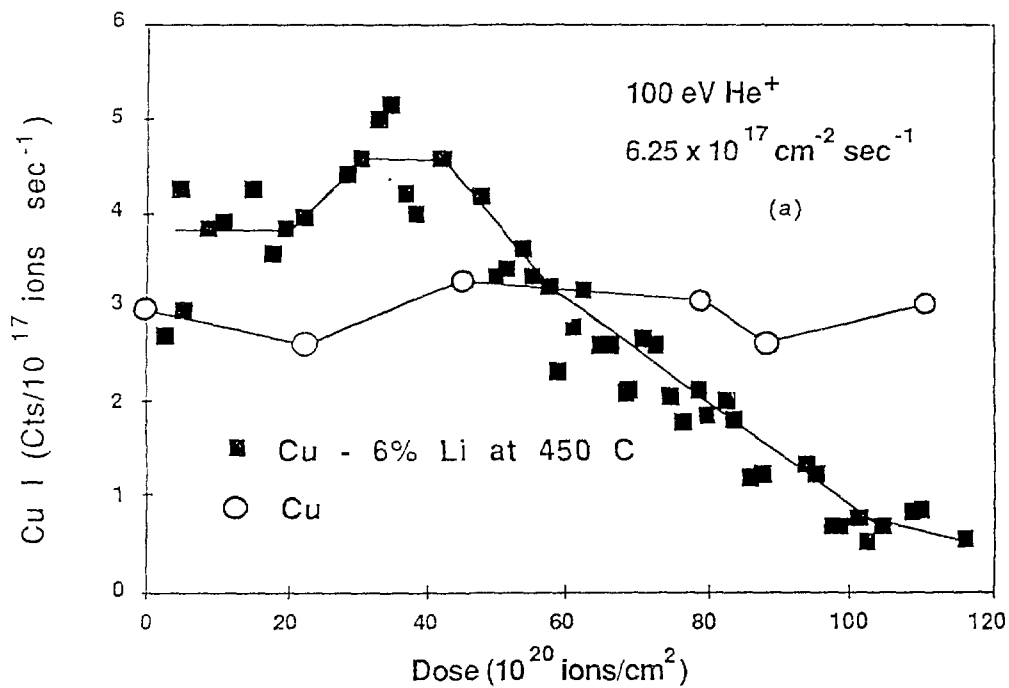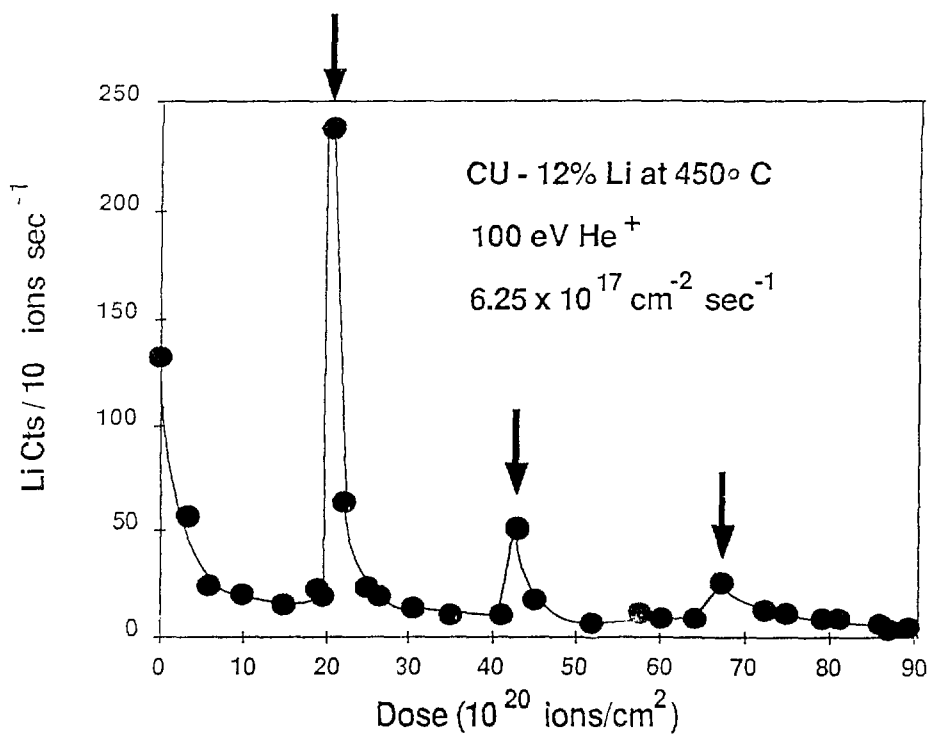
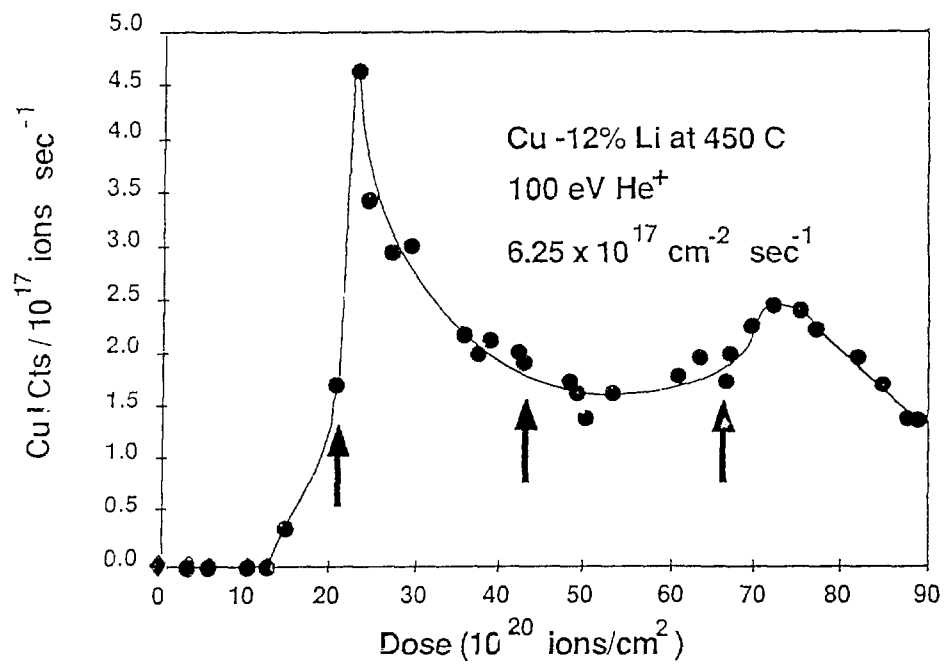COATING GROWTH RATE FOR STEADY-STATE OPERATION (cm/yr)

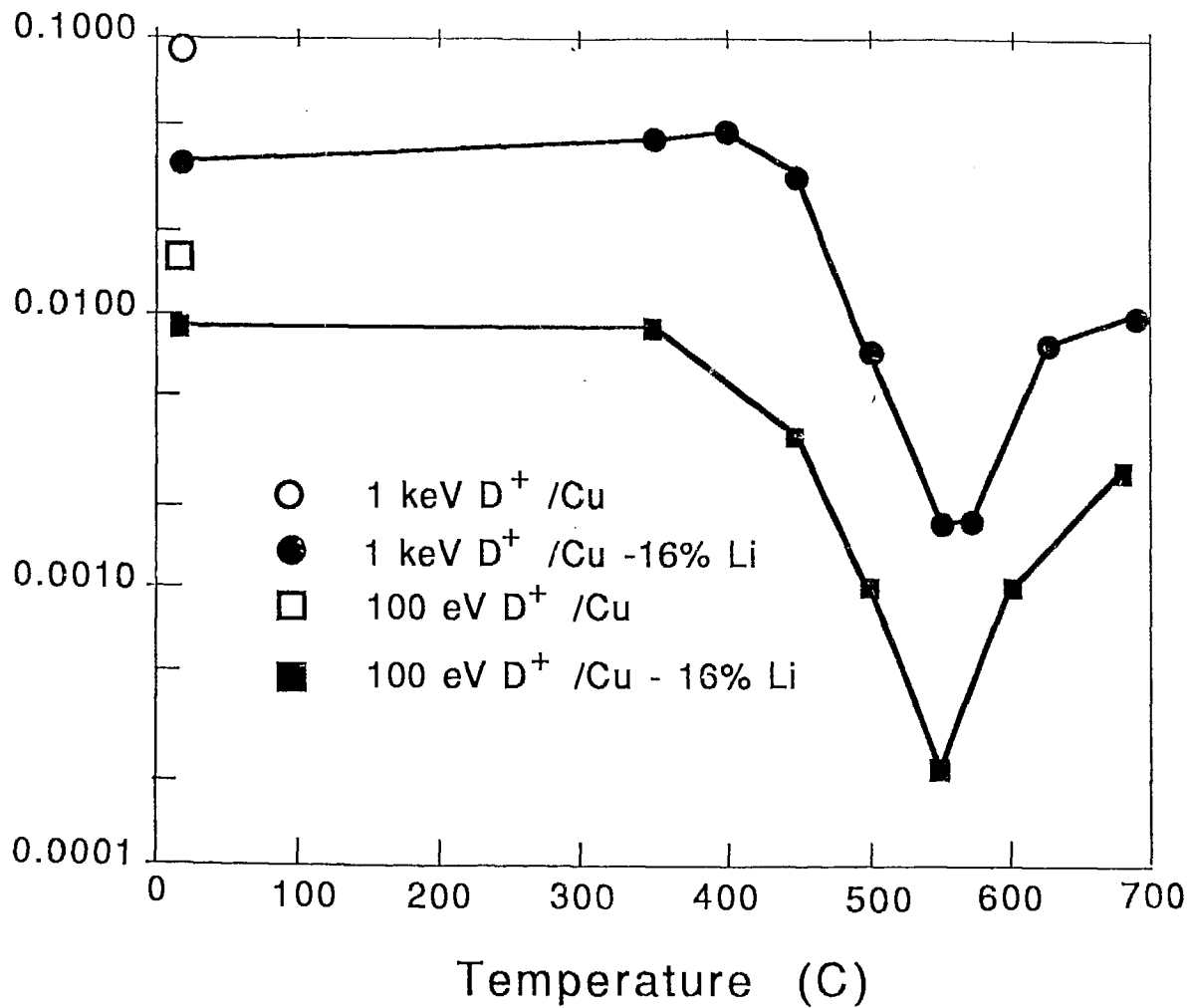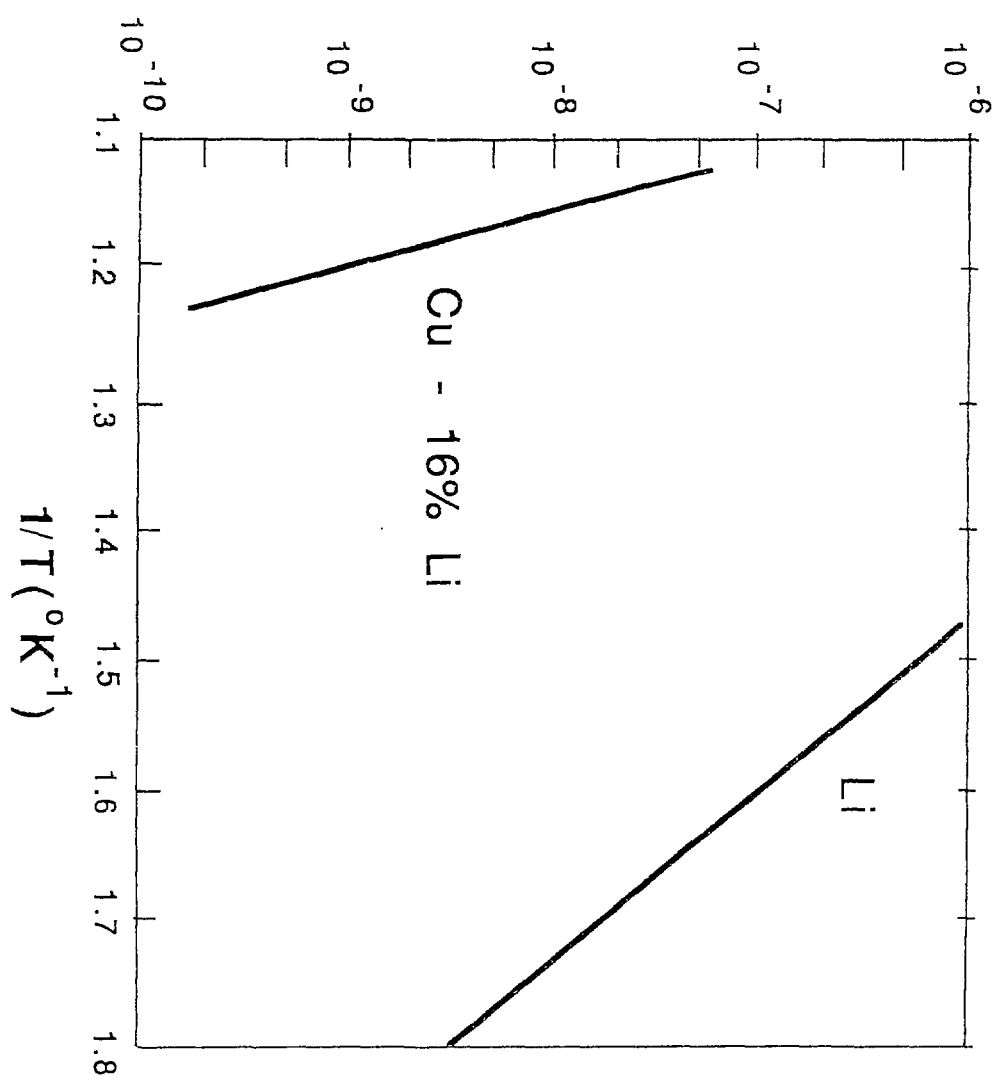BERYLLIUM, 100 eV

TOTAL REDEPOSITION

NET GROWTH RATE (REDEPOSITION MINUS SPUTTERING)

D-T + α SPUTTERING

SELF-SPUTTERING

TOTAL SPUTTERING

LIMITER POINT

Sputtering Yield vs Li thickness (monolayers)

1 keV D$^+$ → Cu-Li
■ Li Yield
□ Cu Yield

200 eV He$^+$ → Cu-Li
● Li Yield
○ Cu Yield

100 eV He$^+$

6.25×10^{17} cm^{-2} sec^{-1}

(a)

Cu $-$ 6% Li at 450 C

Cu

100 eV He$^+$

6.25×10^{17} cm^{-2}sec^{-1}

Cu $-$ 6% Li @ 450 C

Li I (670.8 nm)

(b)

Cu ! Cts / 10^{17} ions sec^{-1} vs Dose (10^{20} ions/cm^2)

Cu -12% Li at 450 C
100 eV He$^+$
6.25 x 10^{17} cm^{-2} sec^{-1}

Li Cts / 10 ions sec^{-1} vs Dose (10^{20} ions/cm^2)

CU - 12% Li at 450° C
100 eV He$^+$
6.25 x 10^{17} cm^{-2} sec^{-1}

Legend:

○ 1 keV D$^+$ /Cu
● 1 keV D$^+$ /Cu -16% Li
□ 100 eV D$^+$ /Cu
■ 100 eV D$^+$ /Cu - 16% Li

Temperature (C)

DISCLAIMER