PHYSICAL REVIEW B VOLUME 22, NUMBER 6 15 SEPTEMBER 1980

Soft-x-ray emission from Li-Al and Li-Mg alloys

J. A. Tagle* and E. T. Arakawa
Health and Safety Research Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee 37830

T. A. Callcott
Health and Safety Research Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee 37830
and Department of Physics, The University of Tennessee, Knoxville, Tennessee 37916
(Received 7 April 1980)

The K-emission band of Li and L_{23}-emission bands of Mg and Al have been measured for Li-Mg and Li-Al prepared in a range of concentrations by either coevaporation or by evaporating thin films of Li on Mg(Al) substrates or Mg(Al) on Li substrates. The Li K spectrum is nearly unchanged at all alloy concentrations in the Li-Mg system but broadens and develops a low-energy shoulder for large Al concentrations in the Li-Al system. Both Mg and Al L_{23} spectra are narrower and more peaked at low energy in the equilibrium alloys than in the pure metals. The "many-body" threshold peaks on the Al and Mg L_{23} spectra are generally suppressed by alloying with Li, except for low-temperature coevaporated Li-Al samples where the peak persists to very Li-rich alloys. Structure studies of these samples are required before the questions raised by these results can be fully resolved.

INTRODUCTION

In the one-electron approximation, soft x-ray emission spectra (SXES) result from transitions between conduction-(or valence-) band states of the solid as a whole and core levels of a single atom in the solid. Thus, in alloys they measure an average transition density of states (TDOS) which samples appropriate angular momentum components of the conduction band in a region localized about one constituent of the alloy system.[1,2] In the dipole transition approximation, K-spectra sample conduction-band states of p symmetry and L-spectra sample conduction-band states of s and d symmetry.

The alloys of light elements were the subject of a number of early SXES experiments.[3-7] In these experiments, it was observed that the TDOS may be different for different components of the alloy, even when the core states have the same angular momentum. For example, careful measurements of the Mg-Al alloy system indicate that the Mg and Al L_{23} spectra have different widths and shapes while the K spectra are nearly identical.[8,9] These experimental results clearly indicate that the old rigid-band model,[10] which predicts identical TDOS for all alloy constituents, cannot be even qualitatively correct. More recently, experimental data have been explained qualitatively in terms of localized densities of states with different TDOS being characteristic of each of the components of the alloy.[1,2,11-14] Sellmyer has recently reviewed the experimental aspects of the electronic structure of metallic alloys.[2]

Recent theoretical investigations of the electronic states in random alloys based on the *ab initio* nonoverlapping muffin-tin-model of the alloy potential function have been reviewed by Ehrenreich and Schwartz and by Gyorffy and Stocks.[14,15] These calculations indicate that, at least in the case of random substitutional alloys, the observed TDOS can be given a satisfactory theoretical explanation.[14-20] It can be shown that it is a good approximation to calculate the transitions occurring near an atom of one constituent of the alloy embedded in a lattice of average potentials.[14,15,17] The average potentials can be specified by one of several prescriptions, the most accurate being the coherent potential approximation. These calculations show that, apart from a matrix element, which can be calculated, the TDOS is directly proportional to a theoretically well-defined local density of states.[17] Detailed calculations have been published that give a good account of the observed Cu-Ni spectra.[18,19] A calculation for dilute alloys of Mg and Al in Li has been published,[20] and additional calculations on the Li-Al and Li-Mg alloys are in progress.[21]

Concentrated alloys often have ordered phases and two-phase regions which introduce complexities that greatly complicate comparison with theory. These problems are particularly severe for the Li_xAl_y systems where two phases exist for about 85% of the possible values of x (Refs. 22-24).

In real metals, many-body effects may modify the SXES spectra, particularly near the Fermi edge.[25] Complete calculations including both band-structure and many-body effects have not been performed for any solid. However, empirical fits to experimental data[26-28] and recent calculations[29,30] indicate that a reasonably good description of the spectra can be obtained by folding a one-electron TDOS with a many-body peaking function that modifies the TDOS near the Fermi edge. The many-body effects are small or absent in the K spectra of Li (Ref. 27), but cause substantial peaking of the L_{23} spectra of Al and Mg (Ref. 28). The many-body threshold peaks have been shown to persist in the Al-Mg alloys.[8] An important result of our study of the Li-Al and Li-Mg systems is that the many-body peaks in the Mg and Al L_{23} spectra are suppressed upon alloying with Li.

In this paper, we report measurements of the K-emission band of Li and of the L_{23}-emission bands of Mg and Al for Li-Mg and Li-Al samples of different composition, prepared either by coevaporation *in situ* or by alternate evaporation of the two alloy components. The data are discussed in terms of the effects of alloying on the one-electron spectra and on the many-body peaks at the Fermi edge.

EXPERIMENTAL PROCEDURES

The emission spectra were measured in an ultrahigh vacuum (UHV) chamber at a pressure of appoximately 2.7×10^{-6}Pa (2×10^{-8} Torr). Samples were prepared by evaporation *in situ* onto a Cu anode cooled by circulating fluid (liquid nitrogen, water, or compressed gas). The substrate temperature was monitored by a thermocouple clamped to the anode surface. A Ti sublimation pump baffle located in the base of the UHV chamber was cooled by liquid nitrogen during the experiments to reduce the contamination of the sample surfaces by water vapor and other condensible gases.

Emission spectra were produced by an 8–10-mA beam of 2.0-kV electrons which bombarded the sample at 45° from normal. A two-meter grazing incidence monochromator fitted with a 1200 lines/mm gold-coated grating and a channeltron detector was used to measure the spectra. Spectra were recorded using 150-μm slits. At this slit width the monochromator resolution was 0.085 ± 0.005 nm in wavelength, which gives energy resolutions of about 0.17 eV at 50 eV (Li and Mg spectra) and of 0.36 eV at 72 eV (Al spectrum). The monochromator resolution was checked by measuring the linewidths of spectra generated by a condensed spark discharge in air.

The output of the channeltron was amplified and fed to a high-speed picoammeter. As the spectra were scanned, the picoammeter output was digitized at 0.08-eV intervals. Five spectra were recorded for each sample and averaged to increase the signal-to-noise ratio. Before plotting, each spectrum was smoothed to remove high-frequency noise, divided by E^5 where E is the photon energy, and normalized to one at the spectral maximum.

The samples were prepared *in situ* by evaporation of the pure metals Al (99.99% purity), Mg (99.99%), and Li (99.9%). The metals were evaporated from Ta boats (Mg and Li) or a tungsten filament (Al). Two different kinds of samples were prepared. For one set of experiments, the evaporation of a thick layer of one metal on a Cu substrate at 300 K was followed by evaporation of a thin layer of a second metal, with the two alloying through a diffusion process. Detailed knowledge of the composition of the alloys in these samples is not available. The evaporation of the second metal was monitored only by observing the decrease of the emission intensity from the substrate metal. We call data from these samples Li on Mg, Mg on Li, Li on Al, and Al on Li (Figs. 4, 5, 7, and 8).

A second set of measurements was carried out on more accurately characterized alloys which were prepared by coevaporation of the two metals of the alloy onto a cooled substrate (85 K). A thickness monitor mounted beside the sample was used to set the evaporation rate of each metal separately, and the samples were then prepared by coevaporation at the preset rates. For concentrated alloys, the resulting emission spectra are substantially the same as those obtained by other workers from alloys of Li-Mg and Li-Al (Refs. 6, 7). The quoted concentration of each material is believed to be accurate within 5%. We call these samples Li-Mg and Li-Al alloys. The data are shown in Figs. 1 and 6. In these figures, the alloys are specified in terms of the atomic percent of each component.

EXPERIMENTAL RESULTS

Li-Mg system

According to the phase diagram for the Li-Mg alloy systems, at 85 K, the alloy forms a solid solution of Li in hcp Mg for concentrations of Li below about 18 at.% and of Mg in bcc Li for concentrations of Mg below about 70 at.% (Ref. 22). We present in Fig. 1 our results for Li-Mg samples prepared by coevaporation onto a substrate cooled by liquid nitrogen to 85 K. The shape of the Li K spectra is essentially unchanged from that of the pure metal, but the L_{23} Mg spectra are greatly modified for Li concentrations greater than about 20 at.%. Over a narrow concentration range, the sharp peak at the Fermi edge disappears and there is a slight enhancement of the broad peak centered at 46 eV.

The presence or absence during evaporation of the electron beam used to excite emission spectra was observed to affect the shape of spectra from coevaporated samples, presumably by supplying

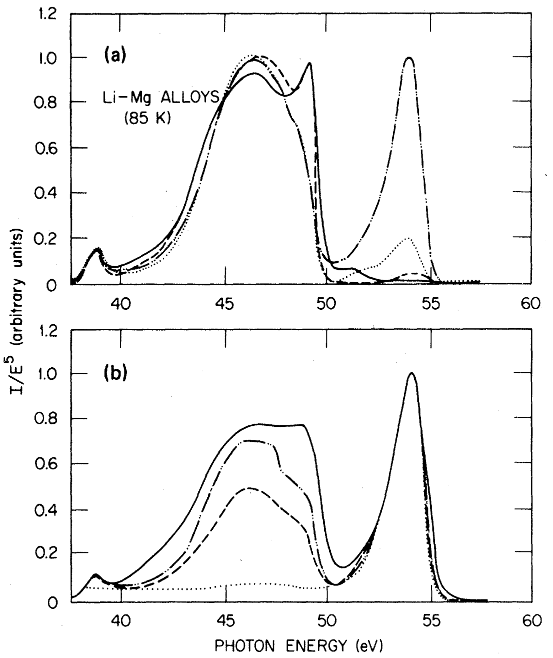

FIG. 1. Emission spectra for Li-Mg alloys. Atomic percent of Mg for the samples are in (a) (——) 100 at. % Mg, (- - -) 89%, (•••) 79%, (–••–) 58%, and in (b) (——) 50%, (–••–) 29%, (– – –) 19%, (•••) 0% (pure Li).

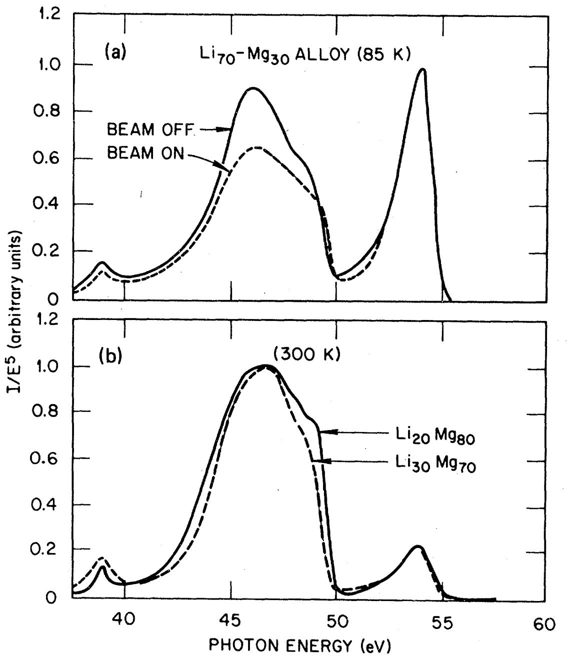

FIG. 2. Li-Mg alloy emission spectra showing the effect of (a) leaving the electron beam on during evaporation and (b) evaporating onto a 300-K substrate.

energy which made the deposited atoms more mobile on the surface. Spectra from samples prepared with and without the electron beam are shown in Fig. 2(a). In this and other samples, the effect was clearly measurable but did not change the qualitative results. In any case, the effect of the electron beam was not studied in detail. All other spectra presented in this paper are from samples prepared with the electron beam turned on.

The substrate temperature is another parameter that may affect the alloy. In Fig. 2(b), spectra are displayed for the alloys $Mg_{79}Li_{21}$ and $Mg_{69}Li_{31}$ prepared by coevaporation onto room-temperature substrates. The spectra are very similar to those from samples prepared on 85-K substrates [Fig. 1(a)]. As will be seen below, a very different result is obtained for Li-Al samples. The Li-Mg samples, however, remain substantially the same, independent of substrate temperature, until the melting and vaporization temperature of Li is exceeded. Then the Li starts to evaporate, raising the pressure in the chamber and shifting the emission results toward spectra characteristic of alloys richer in Mg.

In Fig. 1, a reduction of the Mg L_{23}-emission bandwidth is observed as the concentration of Li increases. In order to compare the bandwidths of the spectra more carefully, the normalized spectra of the Li K spectra and the Mg L spectra are shown in Fig. 3. The reduction of the width of the Mg spectra is seen to be about 0.5 eV, which results mostly from a shift of the Fermi edge to lower energies. This is an important result since Mg may have a sharp peak in its transition density of states at the Fermi level, which a lowering of the Fermi level by 0.5 eV would be expected to eliminate.[31] As for the Li K spectra, very little change in the shape is observed, but there is a small shift of the whole spectrum to higher energies by 0.25 ± 0.10 eV in going from pure Li to the $Mg_{89}Li_{11}$ alloy.

A low-energy edge of the one-electron-Mg emission spectrum can be established for the pure metal by assuming an exponential law for the tailing of the L emission as proposed by Jones.[10] But the true width is difficult to establish precisely, especially in the case of alloys where the tailing of the spectra to lower energies is greater than in the pure metals.

In Fig. 4 we present the spectra obtained from samples prepared by evaporating small amounts of Li onto a fresh, thick evaporated layer of Mg. In this case, as for the Li-Mg samples described above, the Mg emission band narrows, its high-energy edge shifts to lower energies, and the sharp peak on this edge disappears. The narrowing of the spectrum is much more pronounced

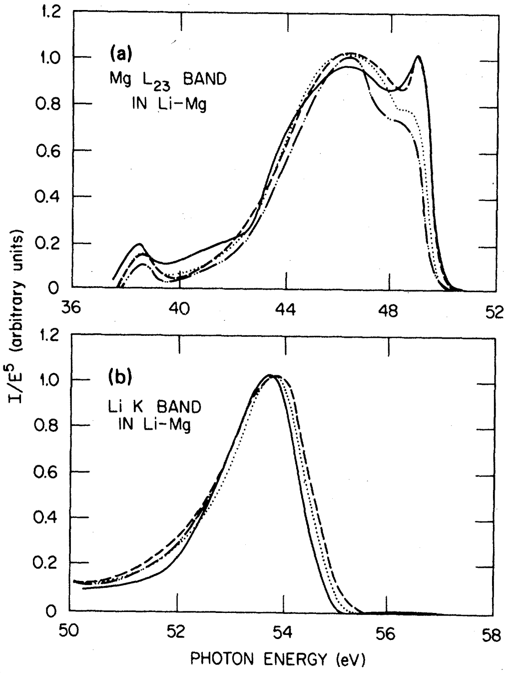

FIG. 3. Normalized Mg L_{23} spectra and Li *K* spectra in Li-Mg alloys. (——) pure metals, (– –) 89 at. % Mg, (• • •) 79% Mg, (– • • –) 29% Mg. (a) Mg L_{23} spectra and (b) Li *K* spectra.

however. In contrast with the results shown in Fig. 3 for the Li-Mg samples, no shift in the energy positions of the *K*-band emission was detected in the Li on Mg samples. Similar results were obtained when layers of Mg were evaporated on thick films of Li. These results are shown in Fig. 5. In all three types of samples, Li-Mg, Li on Mg, and Mg on Li, the spectra are in qualitative agreement with results previously reported for Li-Mg alloys.[6,7]

Li-Al system

In contrast to results for the Li-Mg alloys, there is a strong temperature dependence in the spectra from Li-Al alloys. We find that when the two metals are coevaporated onto an 85-K substrate, emission spectra very similar to those of the pure metals are observed. These data are shown in Fig. 6. For the Al L_{23} spectra, the sharp many-body peak at the high-energy edge remains prominent for all concentrations of Al, though it broadens significantly in the 25 at. % Al alloy. The broad low-energy peak is reduced in relative magnitude, especially in the region near 65 eV. The Li *K* spectrum broadens slightly and develops a weak broad structure centered at about 49 eV on the low-energy side of the spectrum.

When samples are prepared at higher temperatures, very different results are obtained. In Figs. 7 and 8, we show the results obtained from samples prepared by the evaporation of additional small amounts of Al (or Li) onto thick layers of Li (or Al) at 300 K. In Fig. 7, as Al is added to Li, the peak at the high-energy edge of the Al spectrum is decreased to a shoulder, while the broad hump at lower energies narrows and is resolved into a double-peaked structure with maxima at about 65 and 67.5 eV. At the same time, the Li changes from a single-peaked spectrum characteristic of pure Li to a double-peaked spectrum with maxima at 49 and 52.5 eV. Moreover, the more prominent peak broadens, its maximum shifts downward in energy by about 1 eV, and it develops a shoulder on its high-energy edge. For Li on Al samples, the same changes are seen to occur in reverse order [Fig. 8(a)].

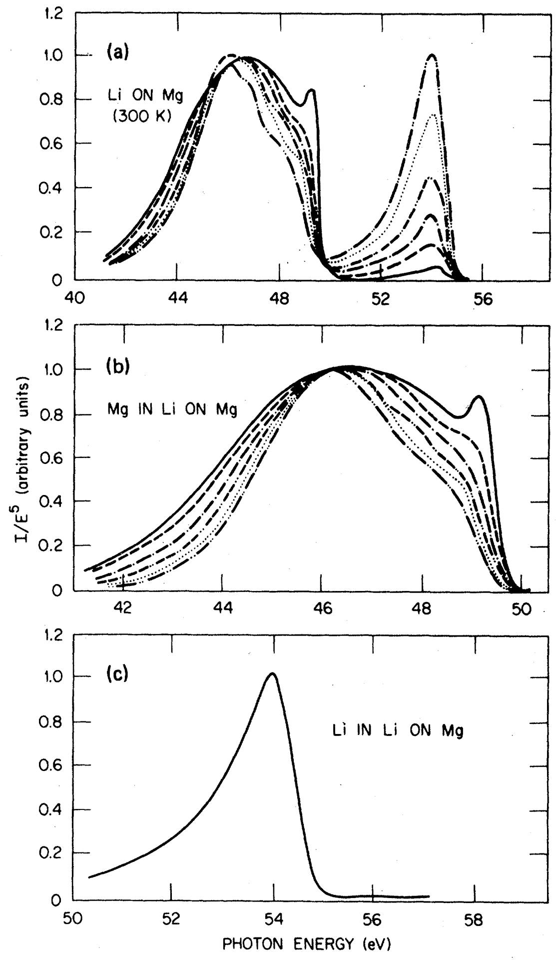

FIG. 4. Emission spectra from Li on Mg samples. (a) Combined spectra, (b) normalized Mg L_{23} spectra, and (c) Li *K* spectra. Curves beginning with (——) and ending with (– • • –) represent samples prepared by evaporating increasing amounts of Li on a thick Mg film.

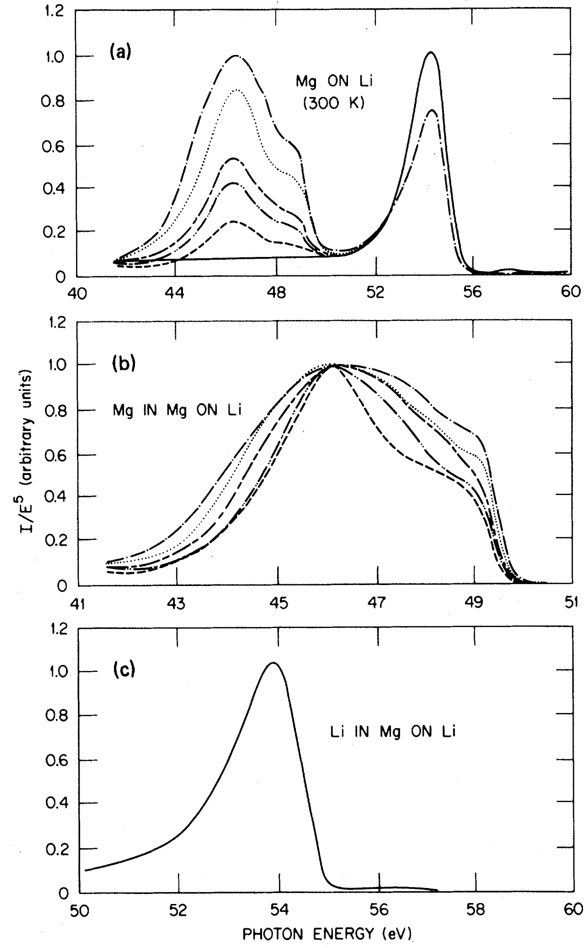

FIG. 5. Emission spectra from Mg on Li samples. Curves beginning with (——) and ending with (–·–) represent samples prepared by evaporating increasing amounts of Mg on a thick Li film. (a) Combined spectra, (b) normalized Mg spectra, and (c) Li spectra.

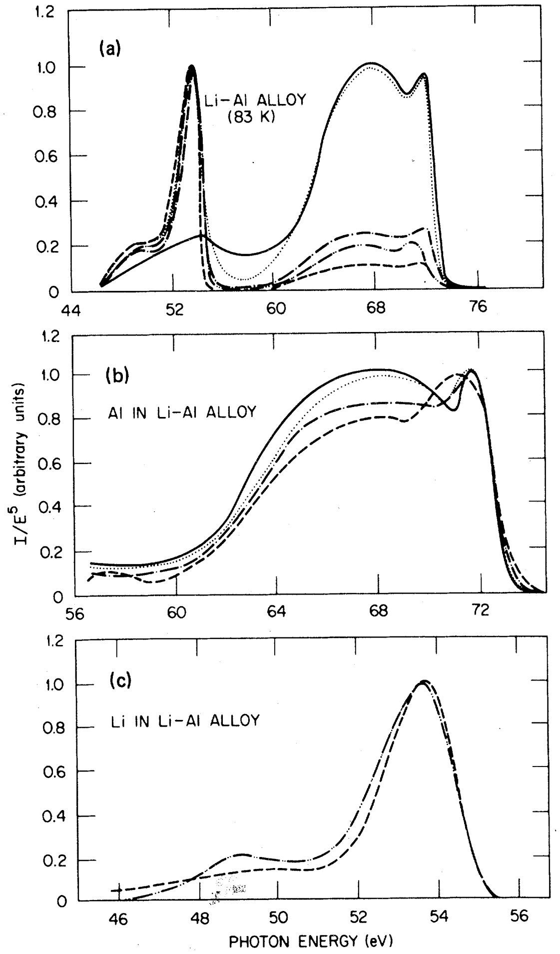

FIG. 6. Emission spectra from Li-Al alloys. The atomic percentage of Al in the alloys shown is (——) 100 at. %, (···) 66%, (–·–) 57%, (–··–) 46%, (– – –) 25%. (a) Combined spectra, (b) normalized Al L_{23} spectra, and (c) normalized Li K spectra.

Very similar results were obtained with coevaporated Li-Al samples if they were prepared on 300-K substrates or if they were raised to 300 K after preparation at 85 K. In Fig. 8(b), the Al L spectrum characteristic of the alloy is produced from a sample prepared at 85 K by raising the temperature very slowly (~2°/min) to 300 K, while the evolution of the spectrum was followed. Over a narrow temperature range near room temperature, the spectrum changed from the characteristic low-temperature spectrum to a spectrum similar to those shown in Figs. 6 and 7. At the same time, the corresponding Li spectrum (not shown) evolves from the single-peaked low-temperature spectrum to a more complex double-peaked spectrum like that shown in Fig. 7(c).

It is known from phase diagrams of the Li-Al system that for most concentrations, the equilibrium alloy is a two-phase alloy in which one or both phases consist of ordered metallic compounds.[22–24] We believe that the results for high-temperature samples represent spectra from near-equilibrium alloys and that the changes in spectra observed when samples prepared at low temperature are raised to 300 K are caused by the formation of the equilibrium alloys via diffusion of Li as the temperature is raised. Evidence that the changes are produced as a result of Li diffusion is provided by the fact that the Li-Al samples could be *rapidly* warmed from 85 K to the vaporization temperature of Li and the Li evaporated without greatly changing the Al spectra in the Al-Li samples.

DISCUSSION

The existence of multiple-ordered and disordered phases in concentrated alloys greatly com-

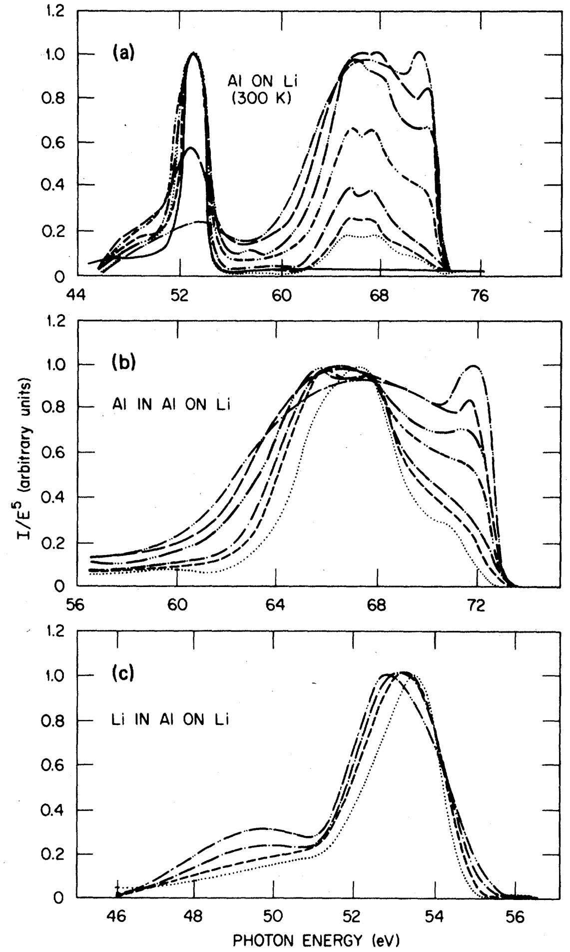

FIG. 7. Emission spectra for Al on Li samples. Curves beginning with (——) and ending with (–••–) represent samples prepared by evaporation of increasing amounts of Al on a thick Li film. (a) Combined spectra, (b) normalized Al spectra, and (c) normalized Li spectra.

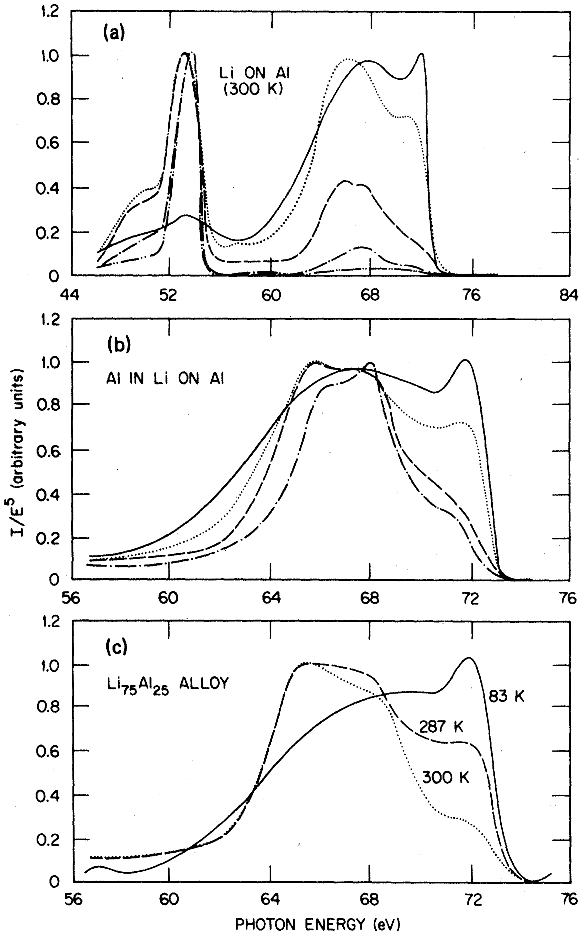

FIG. 8. (a) Emission spectra for Li on Al samples. Curves beginning with (——) and ending with (–••–) represent samples prepared by evaporating increasing amounts of Li on a thick Al film, (b) normalized Al spectra for Li on Al samples, and (c) changes in emission spectra observed when a $Li_{75}Al_{25}$ alloy is warmed from 83 to 300 K.

plicates a discussion of observed SXES structure and renders a detailed discussion impossible if the physical structures are imprecisely known. These problems are minimized in the Mg-Li system since Li_xMg_y forms a random substitutional alloy of hcp symmetry for $0 \leq x \lesssim 18$ atomic percent and a random alloy of bcc symmetry for $30 \lesssim x \leq 100$ atomic percent.[22]

A striking result in the Li-Mg alloys is that the Li *K* spectra remain almost unchanged for all alloy concentrations. A small increase in the width of the spectra for Mg-rich alloys can be explained in terms of a small net increase in the electron density surrounding the Li atom. It is clear from these spectra that the conduction-band states of *p* symmetry are little changed by the alloying process. This implies that extra charge on each Mg ion is almost completely screened within its own lattice cell and that the small screening charge spilling out of the cell affects the Li only by slightly increasing the electron density in the outer region of the Li cells.

The observed changes in the Mg L_{23} spectra are much larger. As discussed above, the peak at the Fermi edge disappears for Li concentrations between 10 and 20 at. % in the coevaporated samples (Figs. 1 and 3). To the extent that this is a DOS effect rather than a many-body effect, it probably results from a change of phase from the hcp "Mg" structure to a bcc "Li" structure.

The detailed shape of the broad low-energy peak of the Mg spectra cannot be discussed in detail without calculations for the concentrated alloys. An important feature, however, is that the spectra narrow for alloys with low concentrations of Mg.

For the coevaporated samples, this narrowing is only about 0.5 eV even for the $Mg_{20}Li_{80}$ alloy. But much greater narrowing is seen in the Li on Mg and Mg on Li samples. This effect may be understood on the basis of the Green's function calculations of Inglesfield for dilute alloys of Mg (and Al) in Li (Ref. 20). His calculations indicate that the Mg spectra should narrow to the width of the Li conduction band and should develop a strong peak at the low-energy edge of the spectra. Results consistent with this prediction are observed in our Li on Mg and Mg on Li samples in spectra representing dilute Mg in Li for each type of sample. In these samples, the spectra are strongly narrowed and a strong low-energy peak appears. The peak to Fermi level energy difference of about 3.0 eV that is observed in the spectra is slightly less than the width of the Li conduction band, in agreement with the Inglesfield calculation. This peak to Fermi level energy separation is a more reliable guide to the total energy width of spectra from isolated Mg in Li than the total width of the experimental spectra because many processes broaden the low-energy edge of alloy spectra. In these samples where concentration gradients must be present, some broadening will be produced by emission from regions of higher Mg concentration. We note finally that the narrowing observed is not primarily a "surface" effect as might be concluded if data were only available for light evaporations of Mg on Li. We find that essentially the same spectra are obtained for thick overcoatings of Li on Mg where the Mg spectra are produced primarily by Mg atoms that have diffused into the Li overlayer.

The interpretation of the Li-Al data is much more problematical, due to lack of adequate knowledge of the alloy structure. We observed that the spectra from low-temperature coevaporated samples (Fig. 6) were similar for alloys in the range from $Li_{20}Al_{80}$ (not shown) to $Li_{75}Al_{25}$. A possible explanation for this result is that the evaporated atoms are mobile on the surface and segregate into separate islands of Li and Al. The similarity of the spectra to "pure-metal" spectra would then be explained by the existence of segregated islands of Li and Al, and the broadening of L spectra observed for dilute concentrations of Al would result when the islands become very small. The large changes in spectra observed at high temperature would then be attributable to phase changes which occur via diffusion to the known phases of the Li-Al alloy system. This interpretation has the virtue of accounting for the persistence of the many-body peak of the Al L spectrum to very Li-rich alloys (at least to $Al_{25}Li_{75}$) but rests on an unproved assumption about the alloy structure.

An alternative hypothesis is that samples coevaporated onto 85-K substrates are frozen into place as random nonequilibrium alloys. We believe that data argue against this interpretation. First, the spectra do not resemble those expected on the basis of either Inglesfield's calculations for Al impurities in Li or of Korringa-Kohn-Rostoker–coherent-potential-approximation calculations for Li-Mg alloys.[20,21] Moreover, it implies that the many-body peak on the Al spectra persists to high Li concentrations in disordered Al-Li samples but disappears when the samples are converted to the equilibrium alloy phases at higher temperature. This conclusion would be hard to reconcile with many-body theory or with the Li-Mg data where the many-body peak is absent for Li concentrations above about 20 at. % even when the equilibrium alloy phase is a random substitutional alloy of Mg and Li that should have properties similar to that of disordered Li-Al samples.

Additional experiments are needed to resolve questions about the low-temperature coevaporated samples. A determination of the alloy structure is particularly important, since a proof of the existence of a random alloy would pose serious questions about the many-body peak in Al.

If we assume the formation of segregated islands of Li and Al for coevaporated samples at low temperature, the most important result obtained in this study may be that the threshold peaks at the edges of both the Al and Mg L_{23} spectra disappear when these metals are alloyed with Li. The accepted interpretation of this peak in Al is that it is the threshold singularity predicted by many-body theory.[25] Thus we conclude that alloying of Al with Li effectively removes the many-body threshold anomaly of the Al L-emission spectrum. The interpretation is more complex for Mg, where the threshold peak has been attributed to both band-structure[31] and many-body effects.[25] A recent attempt to fit both the emission and absorption spectra of Mg suggests, in fact, that both band-structure peaks and many-body threshold anomalies must be used to obtain good fits to the spectral edges.[28,32] The suppression of the threshold peak of Mg by alloying with Li probably will not allow the elimination of either source of the threshold peak, since both sources may very well be removed by alloying. Presumably the many-body peak is suppressed by the same mechanism operating in Al, while the band-structure peak may also be removed when the decrease in electron density causes the Fermi level to move below a sharp peak in the TDOS, or when a structural change in the alloy changes the density of states and eliminates the TDOS peak at the Fermi level.

We believe that the room-temperature Li-Al samples produce spectra more nearly characteristic of the equilibrium alloys. According to published phase diagrams, for $T < 180\,^\circ\mathrm{C}$, $\mathrm{Li}_x\mathrm{Al}_y$ forms single-phase alloys, conventionally designated α, β, γ, δ over narrow concentration ranges. α is a solid solution of Li in Al (0–5 at. % Li), β is nonstochiometric Li-Al (46–56 at. % Li), γ is $\mathrm{Li}_3\mathrm{Al}_2$ (60 at. % Li), and δ is $\mathrm{Li}_9\mathrm{Al}_4$ (69 at. % Li). For other concentrations, two-phase alloys are stable. They are $\alpha+\beta$ (5–46 at. %), $\beta+\gamma$ (56–60 at. %), $\gamma+\delta$ (60–69 at. %), and δ+ Li (69–100 at. %) (Refs. 22–24).

In the absence of either accurate structure determinations or TDOS calculations for the various structures, little interpretation can be made of the spectra from samples with intermediate alloy densities. We note, however, that a characteristic L_{23} spectrum is observed for a wide range of alloy concentrations. It extends from about 62 to 73 eV, has barely resolved low-energy peaks at about 65 and 68 eV and a high-energy shoulder at 72 eV. For more Li-rich alloys, the high-energy shoulder is increasingly suppressed. For very dilute alloys of Al in Li [Fig. 7(b), curve 7; Fig. 8(b), curve 3], the L-spectral width decreases sharply at its low-energy edge and becomes qualitatively very similar to the L_{23} spectra of Mg. Though the spectral width never approaches the width of the Li conduction band, the result is still in qualitative agreement with the Inglesfield result for Al, since he finds that for Al in Li a bound state separates slightly from the conduction band. When thermally broadened, this would appear as a peak in the emission spectrum at an energy separation from the Fermi level slightly larger than the Li conduction bandwidth. This is a plausible explanation, but may be wrong since no account is taken of the spectra that may be produced by the different alloy phase structures.

For dilute Li in Al, the Li K spectrum is greatly modified [Fig. 7(c)]. The high-energy peak broadens and is barely resolved into two peaks, one at the position of the pure metal Li peak (~54 eV) and a second at about 52.5 eV. For higher Li concentrations, the two peaks are unresolved and appear as a broad peak centered somewhere between these two energies. In addition, a broad peak centered at about 49.5 eV appears at the lower edge of the spectrum that doubtless results from the overlap of "Al-like" states into the Li cells. All of these are characteristic features that can be sought in TDOS calculations of Li in Al.

ACKNOWLEDGMENT

This research was sponsored by the Office of Health and Environmental Research, U. S. Department of Energy, under Contract No. W-7405-eng-26 with Union Carbide Corporation.

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[*] Present address: Departamento de Fisica Fundamental and Instituto de Fisica del Estado Solido (SCIC), Universidad Autonoma, Madrid-34, Spain.

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