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THE DETERMINATION OF Mg SURFACE ENRICHMENT IN HEAT TREATED AlMgSi ALLOYS USING THE SXES METHOD

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The heat treatments result Mg loss by diffusion from AlMgSi alloys. This was identified by many authors using different bulk measurements. Our results show, that the Mg concentration essentially increases during the heat treatment near to the surface.

It has been known for quite a time that longer heat treatment of AlMgSi alloys is producing Mg enrichment on the surface[1-4].

Most of the investigations referring to this did not contain concrete data concerning the amount of Mg in the surface layer. Bulk type measurements were performed, with the exception of the[4] measurement, extrapolating on the surface the Mg concentration.

In our present paper we attempted to determine the Mg concentration enriched in the 200 nm thick surface layer.

For the investigation of the surface concentration of Mg we used the soft X-ray emission spectroscopy method /SXES/. We performed the measurements using the transitions of the Al $L_{2,3}$ levels.

On the basis of the calculation of Segall[5] and Rooke[6] was obtained Al $L_{2,3}$ SXES curve, using the[7] band structure calculation.
The symmetry points of the Brillouin zone are : L_2'/68,6 eV/,

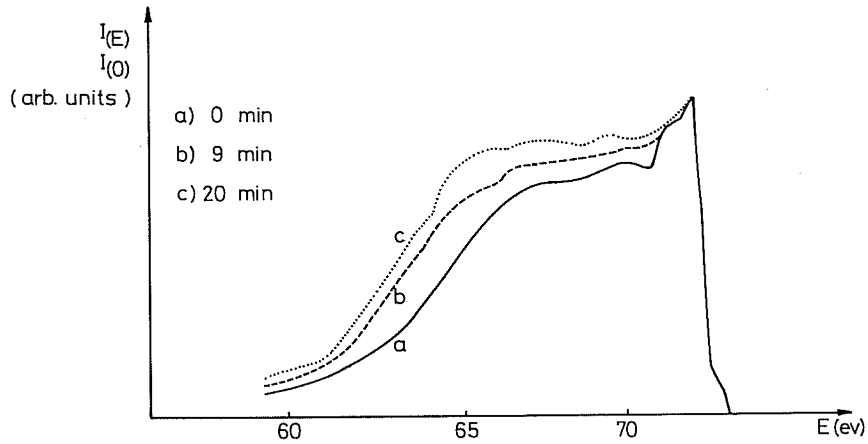


Fig.1 : SXES curves of AlMgSi alloys after annealing in vacuum
a. 0 min
b. 9 min
c. 20 min

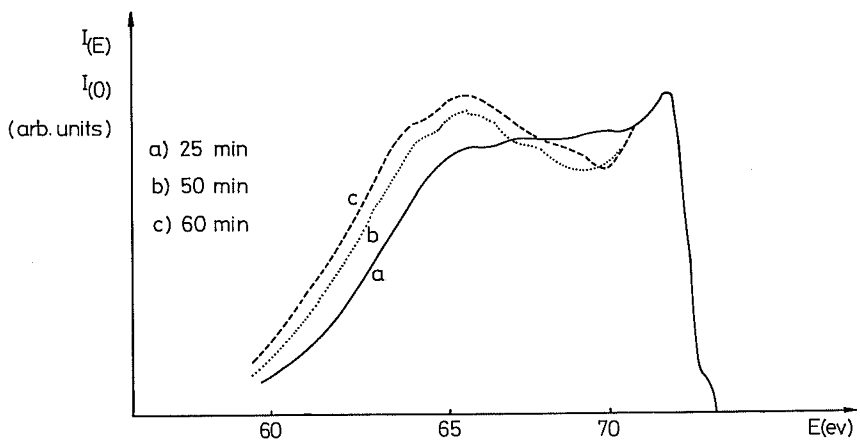


Fig.2 : SXES curves of AlMgSi alloys after annealing in vacuum
a. 25 min
b. 50 min
c. 60 min

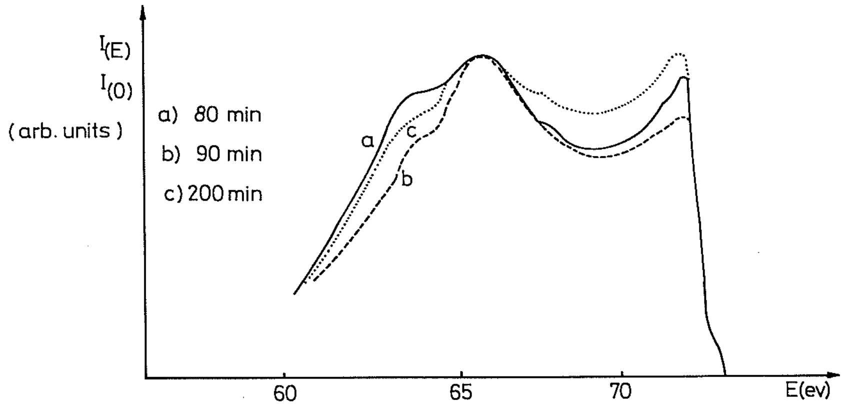


Fig.3 : SXES curves of AlMgSi alloys after annealing in vacuum
a. 80 min
b. 90 min
c. 200 min

L_1/69,0 eV/, X_4/70,1 eV/, X_1/71,3 eV/, K_1/71,4 eV/, W_3/72 eV/, K_1/72,7 eV/. The emission edge characteristic of the Al Fermi surface appears at 72,8 eV. The type of equipment was RSM 500/ Burewestnik, Leningrad/[8]. The resolving power in the used range was 0,07 nm /0,3 eV/. The composition of the specimens was the following : Al matrix, Mg 0,58 wt % /0,65 at %/, Si 0,35 Wt %/, 0,34 at %/,Fe 0,14 wt %/, 0,068 at %/, Cu 0,01 wt %/, Ti 0,03 wt %/.

The specimen was annealed for 45 min at 800 K in air and was quenched in water at room temperature.

The final treatment of the specimens was done "in situ" with an exciting electron beam /4 kV, 0,3 mA/ in a vacuum not worse than 10^{-4} Pa. The temperature of the specimen during this heat treatment was 530 K $\pm$ 30 K. It was measured by the new method[9].

For the determination of the Mg concentration we used H. Neddermayer's measurements[10].

As a starting point and for comparison we used the plot of AlMgSi taken after the annealing and quenching of the specimen /figure 1, curve a/. The curve in its main lines is in agreement with the pure Al $L_{2,3}$ SXES curve, with the exception that the fine structure is less pronounced.

The plot shown in figure 1 curve b was taken after 9 min in situ heat treatment, essential variation has not been

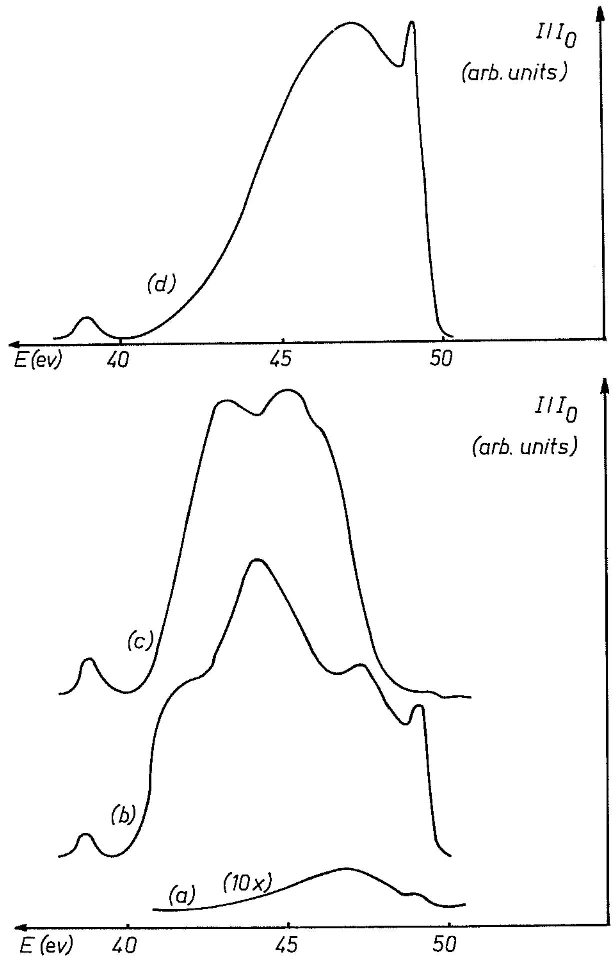
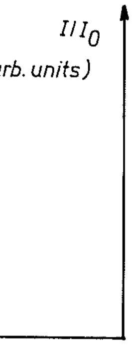


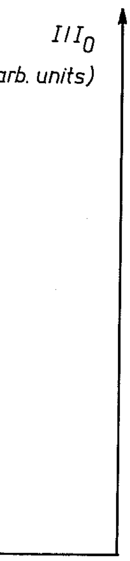
Fig.4 : SXES curves, Mg $L_{2,3}$ peak of AlMg alloy before and after annealing in vacuum

a. before annealing
b. after annealing

found yet. The situation after 20 min is shown in figure 1 curve c on which the appearence of a new local maximum can be observed at 66 eV.

With the further increase of the time of heat treatment, this peak emerges more vigonrously and at the same time the maximum of the large peak below the emission edge gradually decreases. /Fig.2 curves : a,b,c/. After 80 and 90 min a new





loy before and after

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d 90 min a new

local peak appears at 63 eV in figure 3 curves a,b, curve c represents the emission spectrum after 200 min heat treatment, it does not change with further heating. Similar experiments were carried out using Al-5 wt % Mg alloy. The SXES received from the sample without any preliminary heat treatments is in the figure 4 curve a. The next curve /Fig.4, curve b/ was made after 530 K heat treatment for 100 minutes, and the surface was cleaned by electron beam.

On the basis of[10] Neddermayer's work on the various composition of AlMg alloys, the spectra presented on Figure 1 curves a,b,c correspond to 0,6 at %, 10 at % ± 5 %, 20 at % ± 10 % concentration. Our experimental results on figure 2 curves a,b,c correspond to 30 at % ± 10 %, 40 at % ± 10 %, 50 at % ± 10 % concentrations. The emission spectra on figure 3 curves a,b,c correspond to 60 at % ± 10 %, 70 at % ± 10 %, 40 at % ± 10 % of Mg concentrations.

Thus the maximum Mg concentration in the surface layer under given conditions is about 70 %. It is most likely that the Mg atoms are outdiffusing from the matrix, and they evaporate from the surface during the same period.

In accordance with the described results it can be ascertained that due to the effect of heat treatment for longer periods of time /90 min/ at 500 K and higher temperatures, the Mg on the surface layer vigonrously enriches and instead of the 24 % obtained through extrapolation with the[4] measurement, according to our measurements the concentration increases to 70 %.

The behaviour concerning the SXES of the Mg $L_{2,3}$ line has been studied on AlMg alloy so far. According to the SXES measurements low concentration alloy gives a very small effect. The same alloy shows a significance increase of Mg amount about 13 times greater in the surface layer after 100 min. heat treatment at 530 K.

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