TRITIUM RELEASE CHARACTERISTICS OF Al-Li ALLOYS EXAMINED BY LIQUID SCINTILLATION TECHNIQUE
H. Saito, Y. Ishida, H. Yoshida

To cite this version:
H. Saito, Y. Ishida, H. Yoshida. TRITIUM RELEASE CHARACTERISTICS OF Al-Li ALLOYS EXAMINED BY LIQUID SCINTILLATION TECHNIQUE. Journal de Physique Colloques, 1987, 48 (C3), pp.C3-541-C3-545. <10.1051/jphyscol:1987363>. <jpa-00226594>

HAL Id: jpa-00226594
https://hal.archives-ouvertes.fr/jpa-00226594
Submitted on 1 Jan 1987

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L’archive ouverte pluridisciplinaire HAL, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d’enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.
TRITIUM RELEASE CHARACTERISTICS OF \( \text{Al-Li} \) ALLOYS EXAMINED BY LIQUID SCINTILLATION TECHNIQUE

H. SAITO, Y. ISHIDA and H. YOSHIDA*

*Sumitomo Light Metals Ind. Ltd, 3-1-12 Sennen, Minato-ku, Nagoya-shi 455, Japan

University of Tokyo, Institute of Industrial Science, 7-22-1 Roppongi, Minato-ku, Tokyo 106, Japan

Abstract

Hydrogen release characteristics of \( \text{Al-Li} \) alloys at room temperature were examined by liquid scintillation analysis of tritium which has been doped electrolytically in the bulk specimen. The tritium release showed quantitatively that both solubility and mobility of hydrogen in the alloy is strongly functions of lithium concentration in the matrix suggesting the nature of tritium trapping micro-structures.

Experimentals

The \( \text{Al-Li} \) alloys were prepared in the form of a sheet specimen of thickness 1.0 mm by cold rolling. The specimen was sealed in a silica tube containing Ar and were heat-treated as shown in Table 1. After the heat treatment, the specimen was polished electrolytically in nitric acid and methanol solution of volume ratio 1:3 kept at 313K. After polishing the specimen was washed thoroughly with ethanol. The bulk specimen was charged cathodically in tritium water as shown in Fig.2. The electrolysis of tritium was made in 0.002N-Sodium hydroxide aqueous solution. The specific radioactivity of the solution was 0.1 Ci/cm\(^3\). Electric current density and electrolytical charging time was 0.05-5 \( \mu \)A/cm\(^2\) at 7.2Ks respectively.

During the electrolytical tritium charging, helium gas was made to flow at a constant rate and the tritium contained in the gas was oxidized to tritium water in an electric furnace containing heated copper oxide. The tritium water was cooled in the spiral copper pipe and absorbed firstly by magnesium perchlorate, and finally in water. The water, however, didn't show any radioactivity confirming that the tritium leakage to atmosphere was negligible. For safety the whole apparatus was contained in a draft chamber.

After tritium charging the specimen was washed immediately with methanol as shown in Fig.2. The bulk specimen was placed at a hanging platinum wire(0.15\( \times \)X3mm) and immersed in dioxane scintillator of 10cc in volume. Radiation (\( \gamma \)-ray 18KeV) from tritium either in the specimen or diffused out from the specimen to the dioxane scintillator was measured by a liquid scintillation counter(Parkard TRI-CARB-3255). It was measured for 10-20 times at every 30 seconds. The whole sequence was repeated after changing the dioxane scintillator, the tritium counting rate increased with time due to tritium having diffused out from the specimen. The measurement was repeated for three days each time renewing the dioxane scintillator and changing the measurement time until the increase in the tritium counting rate become immesurable. Tritium release rate (cpm/min) was estimated from the initial slope of the counting rate increase just after renewing the dioxane scintillator(1).

Results

Tritium release characteristics of an \( \text{Al-Li} \) alloy was shown Fig.3. Tritium total counting rates increased in time and the increment indicated the release of
diffusive tritium from the specimen. The slope decreased gradually and became zero after three days. Primary tritium counting rates, as shown in Fig.3, correspond to the release rate of tritium upon renewing the dioxane scintillator (1).

1. The effect of heat treatment temperature and lithium concentration.

Tritium release characteristics of Al-Li alloy were analyzed as a function of heat treatment condition and lithium concentration as shown in Fig.4 and 5 respectively. The case of Al-3.1wt.%Li was shown in Fig.4. The tritium release counting rates were high in annealed than in overaged specimens. The values differed by an order of magnitude in the initial release characteristics, but after three days, the two release rates were almost the same.

In the same manner, tritium release characteristics of Al-0.24wt.%Li alloy were compared. The tritium release counting rates of annealed, aged and overaged specimens showed a similar characteristic. The three lines are parallel. The specimen was heat treated at 823K for 7.2Ks and showed highest tritium release counting rates. The lowest values were with a specimen heat treated at 723K for 3.6Ks and overaged 423K for 774Ks.

The effect of lithium concentration to the tritium release is shown in Fig.5. The specimens were treated at 823K for 3.6Ks. The Al-0.24wt.%Li alloy released most gradually while Al-3.1wt.%Li alloy showed an abrupt decrease. The specimen aged at 823K for 3.6Ks and overaged at 423K for 774Ks charged gradually.

2. The effect of charging current density, time and thickness of the specimen

Tritium release in 3.1wt.%Li alloy was affected by the current density as shown in Fig.6 and by charging time and the thickness of the specimen (fig.7) respectively.

Firstly, tritium releases at electric current densities 0.025 and 0.25μA/cm² were compared. When the current density was below 0.025μA/cm², the slope was nil. Therefore, the tritium counting rate decreased gradually. To compare 0.25 and 0.025μA/cm², the counting initial counting rate was high by an order of magnitude for few minutes. Afterwards, the slope showed steep decrease rate. The tritium release differed by the charging time. At the tritium charging time of 0.5hr and 1.0hr nearly the same counting rates were found. In case of 2hrs charging, however, an increase between 2 to 3 times was noticed. In the Al-3.1wt.%Li alloy the effect of the thickness of the specimen was evident as shown in Fig.7. The specimens were marked at one side by manicure paint and charged at 5μA/cm² for 7.2Ks. With increase in the thickness of the Al-Li specimen, tritium release counting rate has increased, until the thickness of 0.58mm was reached.

Discussion

The tritium release counting rates were highest with an annealed specimen than aged and overaged specimens. With increase in the lithium concentration, the tritium release rate increased. In aged and overaged specimens the release characteristics seemed the same because the lines in Fig.7 are parallel in spite of the magnitude differing by an order of magnitude.

The concentration of H+H of the specimen (2,3,4) were estimated from the initial counting rate and counting rate of as charged specimen.

In Table 1 current densities were shown with the concentrations of total H+ H. The effect on the tritium release rate was first analysed by comparing the initial counting rates and the slope measured immediately after tritium charging. With annealed specimen of 3.1wt.%Li specimens the release rate R decreased with time by the same exponent n=2/3 where R=R_t^n . The exponent didn't charge with 0.24wt.%Li specimens. Although the R_t value itself decreased with increase in the aging
time.

The release rate of 3.1 wt.%Li alloy returned basically to that of 0.24 wt.%Li alloy upon aging at 423K. It may reflect the decrease in the matrix concentration of lithium by the precipitation. The remaining small effect of aging time in Al-0.24 wt.%Li alloy and lithium content in the aged specimen may reflect the amount of trapped hydrogen in the changed specimen, because the release exponent n was the same with the specimens.

Conclusion

The present analysis of the release characteristics showed that the enhancement of both the solid solubility and release rate of hydrogen by lithium concentration in the matrix in annealed and water-quenched alloys. The effect of age precipitated Al-Li alloys to the release rate is small in present aged alloys. The analysis suggested that the solid solubility and mobility of mobile hydrogen is a function of lithium concentration in the matrix. The effect of tritium concentration may be neglected in the range of the present experiment.

References

Elaborated time (min)

Fig. 3 Tritium release measurement by a liquid scintillation counter.

Fig. 4 Tritium release characteristics as a function of heat treatment in an Al-3.1wt.%Li alloy.

Fig. 5 Tritium release characteristics of alloys with 3.1 and 0.24wt.%Li concentration. It was heat treated at 823K for 3.6ks.
Fig. 6 Tritium release as a function of the charging current density. It was annealed at 825K for 3.6Ks and aged at 423K for 0.8MKs.

Table 2 The total \( H^+ H \) concentration as a function of electric current density in ppm. Figures in the parenthesis show the tritium counting rates after three days. The specimen were annealed at 823K for 3.6Ks and aged at 423K for 86.4Ks. All specimens were charged for 7.2Ks.

<table>
<thead>
<tr>
<th></th>
<th>0.05 ( \mu A/cm^2 )</th>
<th>0.5 ( \mu A/cm^2 )</th>
<th>5 ( \mu A/cm^2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>pure aluminium</td>
<td>3.95 ppm (7.53)</td>
<td>5.89 ppm (13.11)</td>
<td>10.89 ppm (21.2)</td>
</tr>
<tr>
<td>Al-0.24 wt.%</td>
<td>7.50 ppm (24.90)</td>
<td>8.70 ppm (25.40)</td>
<td>11.3 ppm (37.2)</td>
</tr>
<tr>
<td>Al-3.1 wt.%</td>
<td>10.01 ppm (32.96)</td>
<td>23.4 ppm (79.25)</td>
<td>55.7 ppm (207.0)</td>
</tr>
</tbody>
</table>