MLF Experimental Report

課題番号 Project No. 2016A0024	提出日 Date of Report
実験課題名 Title of experiment Vacancy and hydrogen behavior in Al-Mg-Zn alloys	装置責任者 Name of responsible person Yasuhiro Miyake
実験責任者名 Name of principal investigator Katsuhiko Nishimura	装置名 Name of Instrument/（BL No.） D1
所属 Affiliation University of Toyama	実施日 Date of Experiment January 17 – January 19 (2 days)

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。（適宜、図表添付のこと）
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

1. Al - 3.4 at.% Zn - 1.9 at.% Mg heat treated at 753 K for 1000 minutes and quenched into ice-water on January 14 (four days before the measurement). (noted as Al-Zn-Mg AQ-4d)
2. Al - 3.4 at.% Zn - 1.9 at.% Mg heat treated at 423 K for 400 minutes. (Al-Zn-Mg 150C)
3. Al of a purity 99.99% (base Al); which was used to make Al-Zn-Mg alloys.
4. hydrogen charged Al of a purity 99.99 % by the plasma charging method (Al-H)

*5. Al - 3.4 at.% Zn - 1.9 at.% Mg heat treated at 753 K for 1 hour and quenched into ice-water (1 hour before measurement). (Al-Zn-Mg AQ)

*6. Al of a purity 99.99% heat treated at 848 K for 1 hour and quenched into ice-water. (base Al AQ)

2. 実験方法及び結果（実験がうまくいかなかった場合、その理由を記述してください。）

Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

All the samples were mounted on the spectrometer at D1 beam line. After evacuating the sample space, we measured the temperature dependence of zero-field spin relaxation spectra increasing temperature from 10 K, then 20K to 300 K with a step of 20 K, accumulating 15 mega events at each temperature. TF spectrum with a field of 20 G was measured for a period of 3 mega vents at 10 K. Due to the limited beam intensity, thus a relatively low counting rate, we decided to acquire basic data sets of zero-field spin relaxation for Al-Zn-Mg alloy with the samples of Al-Zn-Mg AQ-4d, Al-Zn-Mg 150C, base Al and Al-H. The measurements with these four samples were completed. However, the last two samples, Al-Zn-Mg AQ and base Al AQ, which would show non-equilibrium state of vacancy, were not explored this time.

The observed zero-field muon spin relaxation spectra were shown in the Figs. 1 for Al-Zn-Mg alloys and 2 for base Al and hydrogen charged base Al. In Fig. 1, it is obvious that muon were trapped in certain sites of both Al-Zn-Mg samples at 20 K since the spectra showed a typical shape of the Kubo-Toyabe function. At the elevated temperature of 140 K, however, there is a clear difference on the relaxation spectra. Muon spins in the Al-Zn-Mg AQ-4d sample relaxed faster than those in the Al-Zn-Mg 150C. The density of the trapping sites of

2. 実験方法及び結果（つづき） Experimental method and results (continued)

Al-Zn-Mg is considered to be much higher than the number of muon particles, so the difference of the relaxation rate at this temperature is possibly due to the difference of micro structures of Zn/Mg/vacancy clusters.

A pure aluminum is known to be quite transparent to positive muons. It is also known that a muon trapping rate is quite sensitive to impurities and defects. The present base aluminum, which was used to make Al-Zn-Mg alloys, has a purity of 99.99 %, which means there existing about 100 ppm impurities. Fig. 2 shows muon spin relaxations in the base Al sample at 200 K due to the impurities and defects. This kind of relaxation behavior, however, almost disappeared at 280 K. The hydrogen charged aluminum sample, Al-H, indicates the faster spin relaxations than those of the base aluminum, thus there are some effects on the spin relaxation originated in the charged hydrogen.

The data analysis has been done following the similar procedure in our previous studies. All the observed spectra were compared with relaxation functions built by a Monte Carlo simulation, in which four variable parameters were employed: the dipolar width (Δ), trapping rate (ν_t), detrapping rate (ν_d), and fraction of initially trapped muons (p_0) [S. Wenner et al.; Phys. Rev. B86, 104201 (2012)]. An ensemble of 60 million muons are simulated to produce a five dimensional relaxation function $f(\Delta, \nu_t, \nu_d, p_0, time)$ [E. Sato, et al.; Hyper. Inter. 17-19, 203-210 (1984)]. Fig. 3 shows examples of the data analysis of the trapping rates deduced from the spectra obtained with Al-Zn-Mg samples. The temperature dependences of the trapping rates of the two samples are different between 40 and 120 K. We made an attempt to estimate an trapping potential energy of Zn/Mg/vacancy clusters, assuming relations: $\nu_d / (\nu_d + \nu_t) = \nu_{d0}\exp(-Q/kT)$ (Q: trapping potential energy). A least-square fit of the data yielded the Q value of 5.2 (+-0.6) meV for Al-Zn-Mg 150C, and 4.6 (+-0.2) meV for Al-Zn-Mg AQ-4d. These values are a bit larger than that of 3.6 (+-0.4) meV obtained in Al-Mg-Si alloys [K. Nishimura et al.; J Phys. Conf. Ser. 551, 012031(2014)].

Fig. 4 shows the extracted values of the dipole widths for the base and hydrogen charged aluminum samples. There is no significant differences at low and medium temperatures, but there is a clear difference above 220 K. At this temperature region, muons are mostly trapped by deep potential sites produced by vacancies. This result implies, therefore, that the charged hydrogens also trapped near vacancies, which increased dipole fields acting on the muons.

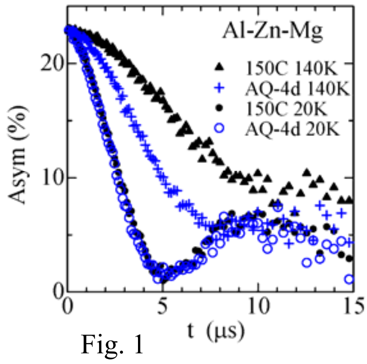

Fig. 1

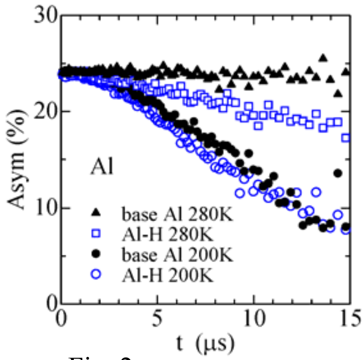

Fig. 2

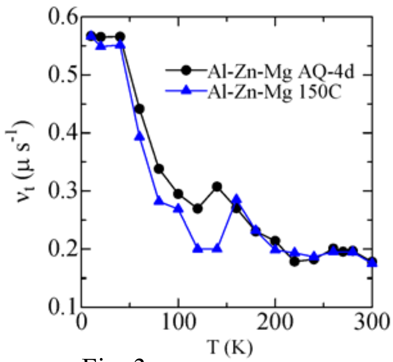

Fig. 3

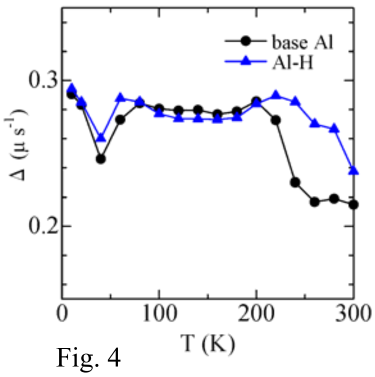

Fig. 4