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Positron annihilation study of the hardening behavior in Al-Cu based alloy by electron and heavy ion irradiation

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Abstract. Al-Cu based alloy, which is generally called duralumin (JIS2017), was irradiated with 10 MeV Iodine ions, 200 MeV Xenon ions and 3 MeV electrons at room temperature respectively. The micro Vicker's hardness and positron annihilation coincidence Doppler broadening (CDB) measurements have been performed before and after irradiation. Only in the case of ion irradiation, the Vicker's hardness increases with increasing ion dose. Nevertheless, there was no difference in the profile CDB spectrum for before and after irradiation. On the other hand, we found that the micro hardness of this alloy, which was Xe ion irradiated and subsequently annealed at 423 K, is greater than that of age hardened alloy without irradiation. CDB ratio curve of the age hardened Duralumin is clearly different in the electron momentum range around 0.015-0.025 mc from that of the ion irradiated alloy. The results of three-dimensional atom probe (3DAP) also show that a lot of small clusters were found after ion irradiation but large precipitations have found in annealed Duralumin. These results reveal that a number of small clusters formed in this alloy after ion irradiation, and they should strongly affects the micro hardness.

1. Introduction

Radiation enhanced segregation is one of the important phenomena. For instance, it is well known that the precipitation of Cu impurity atoms in reactor pressurized vessel (RPV) steels causes their low temperature embrittlement because of the formation of fine Cu precipitates by which vacancy mechanism act as obstacle against the dislocation motions.[1-6] In other words, radiation induced precipitation formation by using supersaturated impurity atoms changes its mechanical property.[7] In the case of RPV, this is a negative effect but we start applying this phenomenon a tool for the hardness control of any alloys.

In the present study, we have tried to irradiate of high energetic particles to Al-Cu based alloy system, which is called Duralmin, in order to control and improve the mechanical properties. Moreover, we have studied the process of some clusters formation in this alloy under electron and swift heavy ion irradiation by using positron annihilation and other techniques. In this paper, therefore, we report the results of the micro Vicker's hardness, positron annihilation Doppler broadening and three-dimensional atom probe measurements for Al-Cu based alloys aged or irradiated with electron and heavy ion, such as Xenon and Iodine.

2. Experimental procedure

Al-Cu based alloy (Duralumin-JIS2017: The chemical compositions of the two alloy samples in the present study are listed in Table 1) was prepared by arc melt method. 10 mm x 10 mm x 1mm size samples were cut for micro Vickers and positron annihilation measurements. They were annealed at 793 K for 1 hour in air and quenched into ice water. I-ion and Xe-ion irradiations with an energy of 10 MeV and 200 MeV into Al-Cu based alloys were performed at room temperature by using a tandem type accelerator at the Japanese Atomic Energy Research Agency (JAEA) Takasaki, JAPAN. 3 MeV electrons irradiation have been performed for Al-Cu based alloy at room temperature by using tandem accelerator at JAEA Tokai. The maximum damage depth for 10 MeV Iodine and 200 MeV Xenon into Al-Cu based alloy was calculated to be approximately 4 and 20 μm, respectively. After irradiation isothermal annealing at 423 and 453 K and isochronal annealing were performed. Coincidence Doppler broadening (CDB) measurement for each annealed and irradiated sample was performed. CDB spectra were measured using two HP-Ge detectors, which were located at an angle of 180 degree relative to each other, and total counts of CDB spectra consist of about more than 10^8. As a positron source, we used $^{22}NaCl$ with an activity of 170 kBq, which was sandwiched by thin Kapton foils. Micro Vickers hardness was also measured by conventional equipment. The measuring temperature was room temperature and the applied load were 25 and 100 gf with holding time of 10 s. Applied load of these value is optimized for estimated damage peak for each ion irradiation. Atom probe analyses were performed for as prepared, aged at 423 K and I-ion irradiated samples using a three-dimensional atom probe (3DAP) equipment [8]. Needle-like atom probe specimens were prepared with a diameter of 100 nm by the micro-polishing technique.

Table 1. Chemical composition of the Al-Cu based alloy.

Cu	Mn	Mg	Si	Fe	Zn	Cr	Al
3.5-4.5	0.4-1.0	0.4-0.8	0.2-0.8	0.7	0.25	0.1	balance

(at.%)

3. Results and Discussions

Micro Vickers hardness of Al-Cu based alloy at room temperature is about 140 Hv. Age hardening of Al-Cu alloy at room temperature is well known generally, but this phenomenon was not observed for this Al-Cu based Duralmin alloy. Figure 1 shows micro Vickers hardness for Al-Cu based alloy before and after 3 MeV electron irradiation up to dose of 1.5 x 10^{18} /cm^2. This figure clearly shows that hardness does not change at all by any electron irradiation dose. Figure 2 shows

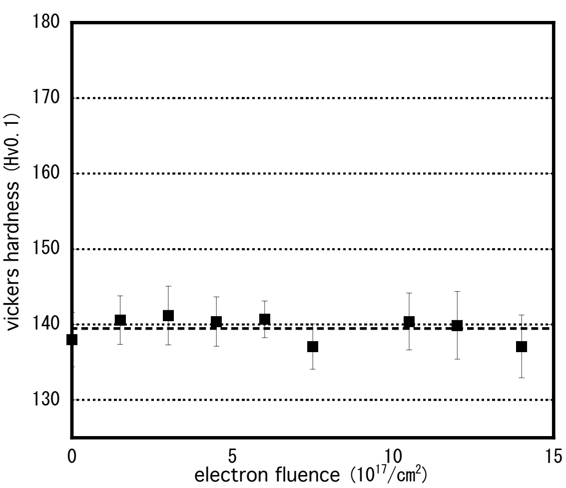

Figure 1. Change in micro Vickers hardness for Al-Cu alloy as a function of 3 MeV electron irradiation dose. (applied load is 100 gf)

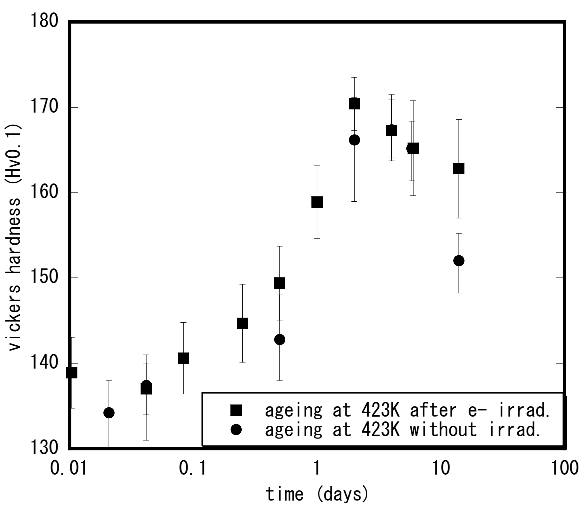

Figure 2. Change in micro Vickers hardness for Al-Cu alloy as a function of dose of 10 MeV I-ion irradiation.

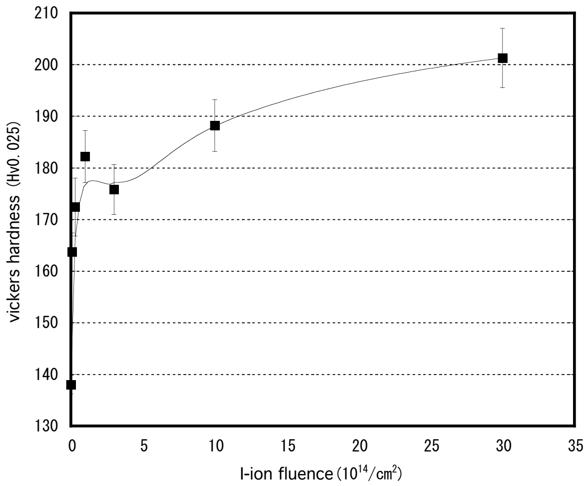

Figure 3. Change in micro Vickers hardness for Al-Cu based alloy as a function of 10 MeV I-ion irradiation dose. (applied load is 25 gf)

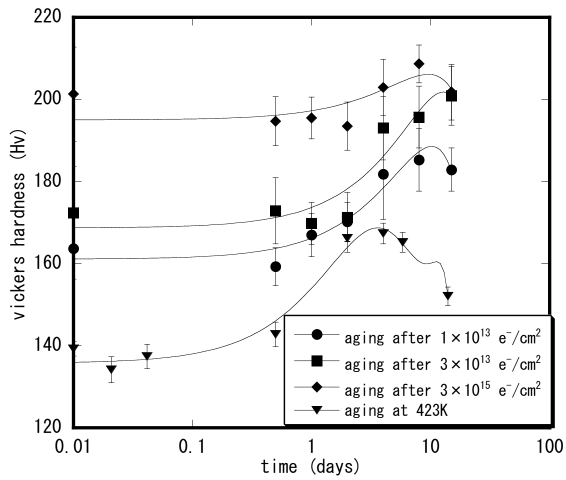

Figure 4. Change in micro Vickers hardness as a function of annealing time at 423 K for I-ion irradiated and unirradiated Al-Cu alloys.

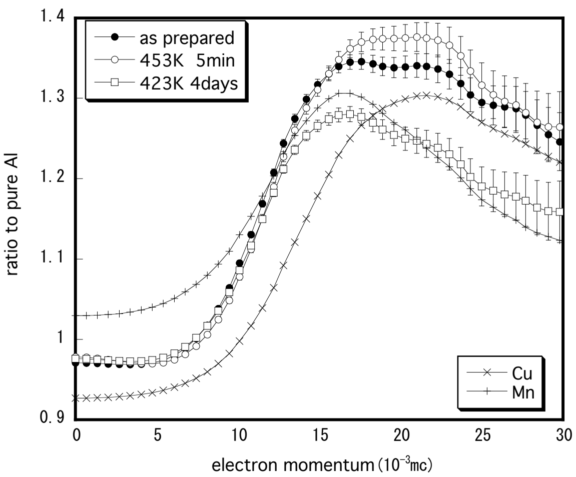

Figure 5. CDB ratio spectra for as prepared and annealed Al-Cu alloys without irradiation in the form of the ratio to pure Al as a function of electron momentum. Also shown is CDB ratio spectrum of pure Cu and Mn normalized to Al.

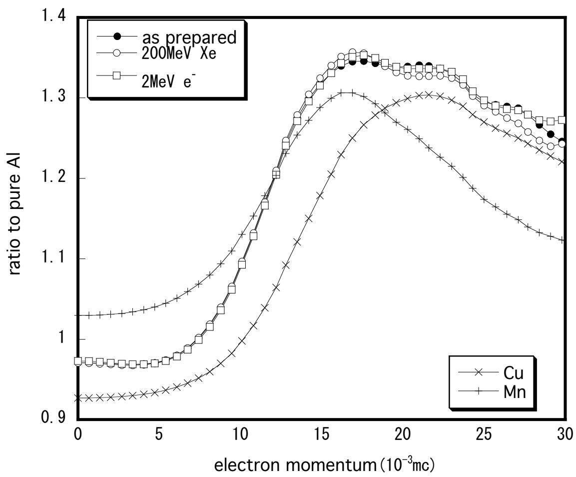

Figure 6. CDB ratio spectra for as prepared, 2 MeV electron and 200 MeV Xe-ion irradiated Al-Cu alloys as a function of electron momentum.

change in hardness with annealing before and after 10^{18} e^-/cm^2 electron irradiation. After electron irradiation, hardness gradually increases with increasing annealing time as same as the aging behavior without irradiation, as shown in this figure. As the result, electron irradiation does not affect hardness.

Figure 3 shows Vickers hardness for annealing after I-ion irradiation. In this figure, Vickers hardness of this alloy increases by I-ion irradiation depending on irradiation dose. The hardness of 200 MeV Xe-ion irradiated sample was also increased. The trend of change in hardness with annealing after ion irradiation is similar to that for age hardening as shown in figure 4. However, it clearly shows that the peak value of hardness after about 10 days annealing for each irradiated sample is greater than that for age hardened sample without irradiation. The indent-depth of the Vickers' load of 25 gf is about few μm and the effect of indentation extends to the depth several times larger than the indent-depth, and the depth of damage peak introduced by this irradiation is about 4μm. Therefore, measured hardness of ion irradiated Al-Cu based alloy is mean value from the surface to around damage peak. In other wards, the local hardness around damage peak might be higher than that of measured Vickers hardness. It is considered that the difference of hardness change between electron and heavy ion irradiation is mainly attribute to total amounts of dpa (displacements par atom). In fact that total dpa of electron irradiation is few orders (at least five orders at damage peak in this case) smaller than that of ion irradiation in this case. However, it is difficult to discuss the dpa dependence of hardness, because the ion irradiation induced dpa for the depth is not unique.

Figure 5 shows the CDB ratio curves for as prepared and age annealed Al-Cu based alloys, which is expressed in the form of the ratio of CDB intensity to that of pure Al. The fraction of Cu and Mn atoms for positron trapping clearly changes by the each annealing. This is supposed that some kind of new clusters are formed and/or grows larger. The results of three-dimensional atom probe measurement shows that some shape of large size precipitates (over 20 nm each), such as Mg and Si rich cluster and Al and Cu rich clusters, are observed only in case of aging without irradiation. Positron trapping at Ω precipitates including Ag and Mg atoms associated with Cu atoms in similar alloy systems is reported [9]. Therefore, certain kinds of these clusters are detected by positron in this case. The CDB ratio profiles for electron, Xe-ion irradiated and as prepared Al-Cu based alloys without irradiation are shown in figure 6. In this figure, no marked change among these samples was observed. In case of electron irradiation, this result has a good correlation with the result of unchanging trend of micro hardness after electron irradiation as mentioned above. Taking into account for these results, it might form small clusters, which are not able to detect by positron and have no effect for hardness, or no cluster formation occur in this alloy matrix by the electron irradiation. On the other hand, ion irradiation makes hardened this alloy even though no remarkable change in CDB ratio curve is observed. The estimated maximum depth of damaged area by this irradiation is about 20 μm and the positron penetration depth as a probe of defect detection is about 100 μm. Therefore, about 10 ~ 20% of positrons annihilate at the damaged area so that formed clusters by the ion irradiation may be extremely small. 3DAP result shows that large size precipitates, which are seen in age hardening heat treated alloy without irradiation, and well known plane type precipitates like a GP-zone in this alloy system are not found in case ion irradiated sample. Instead, large amounts of small size of the order of 1 nm segregations are observed in the ion irradiated sample. Hence, this fact of these small clusters formation after heavy ion irradiation is consistent with the result of CDB measurement. Moreover, 3DAP result also shows that the number density of the cluster in ions irradiated sample is greater than that in age hardening treated one without irradiation. As a consequence, large amounts of small clusters are formed in Al-Cu based alloy by swift heavy ion irradiation, and they should strongly affects the micro hardness.

4. Summary

Al-Cu based alloys of Duralmin were irradiated with electron, Xe and I ions at ambient temperature. We have found that the electron irradiation does not affect the hardness but the change in hardness due to Xe and I-ion irradiation was measured. The change in hardness with annealing at 423 K observed in Al-Cu based alloy irradiated with I ions is remarkably larger than that for age hardened Al-Cu based alloy without irradiation. From the results of positron annihilation CDB and 3DAP measurements, it was found that low density of large size precipitates are formed in aged sample without irradiation, and large amounts of small size clusters are formed in ion irradiated sample, respectively. These facts suggest that these clusters affect the mechanical properties especially the hardness and their effects strongly depend on the size and density of clusters. The present experimental results show that swift heavy ion irradiation can be used for the modification of hardness and its hardness can be improved near surface than in age hardenable Al-Cu based alloy.

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