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# *Effect of Surface and Grain Boundary on the Reversion of Age-Hardened Al-15mass%Zn Alloy*

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SYNOPSIS

Reversion of the age-hardened Al-15mass%Zn alloy, in which ellipsoidal GP zones were formed, was investigated by hardness test. Ellipsoidal zones were reverted more quickly near the surface and grain boundary than in the interior, as spherical zones did. The results confirm their role as sources for vacancies in reversion.

## 1. INTRODUCTION

Age hardening of Al-Zn alloy after quenching develops inhomogeneously due to the effect of surface as a vacancy sink and grain boundary as a easy path.(1) It was found that, when age hardened Al-10mass%Zn alloy was annealed at high temperatures, the reversion started near the surface and delayed in the interior.(2) The fact was explained also by the effect of the surface, but in this case as a source of vacancies.

When Al-10mass%Zn alloy was aged at 293K after quenching from 723K or higher, as in the previous experiment,(2) GP zones formed were small and spherical, which was easily dissolved again on reversion. It is known that large ellipsoidal GP zones are formed in Al-Zn alloys more concentrated in Zn and aged after quenching from

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temperatures lower than 650K.[3] These zones are considered to be stabilized by the shape change.

This paper deals with the effect of the surface on the reversion of the large ellipsoidal GP zones, investigated by hardness test and confirms the role of surface as a vacancy source on reversion.

## 2. EXPERIMENTAL PROCEDURES

Pure aluminum, 99.996%, and pure zinc, 99.999%, in the nominal composition of 15mass%Zn, were melted in a high-alumina crucible in the air. Ingots, 15mm in diameter and about 150mm in length, were homogenized for 180ks at 723K. After peeling, they were hot-forged repeatedly at around 723K to plates of 5mm in thickness. The plates were cold-rolled several times with appropriate intermediate annealing at 723K to plates of 1mm thickness. Grains were coarsened by the strain-annealing method to about 5mm in diameter.

Quenching was carried out in the same way as reported[4] and aging was done in ethyl alcohol at 293K±0.5K. Reversion was studied both by the isochronal and isothermal annealing in ethyl alcohol or silicon oil. In the isochronal annealing experiment, the specimen was annealed at every ten degrees for 0.6ks and hardness was measured after each annealing.

Hardness was measured by Vickers microhardness tester (Akashi, MVK-E) and ultra-microhardness tester (Akashi, MZT-1). In order to examine the variation with depth, hardness test was done at various loads from 0.001 to 4.9N. To the same purpose surface layer was removed by electropolishing and then hardness was measured. Electropolishing was carried out in the perchloric acid-ethyl alcohol (1:4) solution at 20V of voltage and 500A/$m^2$ of current density.

## 3. RESULTS AND DISCUSSION

Fig.1 shows variation with the indentation load, of the hardness of the alloy aged for 120ks at 293K after quenching from 723K. This heat treatment produces spherical GP zones in the alloy.[3] As-aged specimen showed constant hardness from 4.9N down to 0.98N, but at lower loads hardness decreased with decreasing load. This suggests the existence of soft surface layer. After the surface layer 30$\mu$m

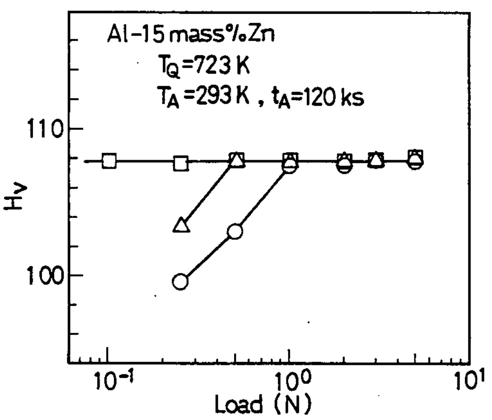


Fig.1 Variation with the thickness of surface layer removed, of the dependence of hardness on the indentation load, for the specimen aged for 120ks at 293K after quenching from 723K. Removed thickness: ○ 0 μm, △ 30 μm, □ 75 μm.

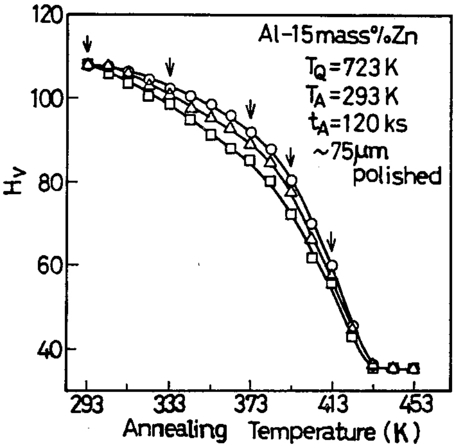


Fig.2 Variation of the isochronal annealing curve with the position of indentation for the specimen aged as in Fig.1 and electropolished to remove 75 μm thickness of surface layer. Indentation load was 0.49N. ○ far from GB, △ near GB, □ on GB.

in thickness was removed, constant hardness was obtained down to 0.49N, but the soft surface layer remained. When thickness of 75μm was removed hardness never decreased down to the smallest load used in this experiment, 0.001N. These results indicate that the thickness of the soft surface layer after aging was less than 75μm.

The specimen aged for 120ks at 293K and electropolished by 75μm, in which soft surface layer no longer existed, was annealed isochronally. Fig.2 shows variation of the isochronal curves in hardness at 0.49N of load with the position of indentation in the grain. The hardness near the grain boundary (abbreviated hereafter by GB), about 70μm distant from GB, fell more rapidly than that far apart, more than 200μm distant, from GB, and that at the GB still more rapidly. At the stages marked by arrows in this figure hardness at the position far from GB was measured at various loads. The results are shown in Fig.3. Although the hardness was constant irrespective of the load before the isochronal annealing, those measured after 333K annealing were lowered from the constant value at the load less than 0.98N. This indicates that the reversion began at the surface but

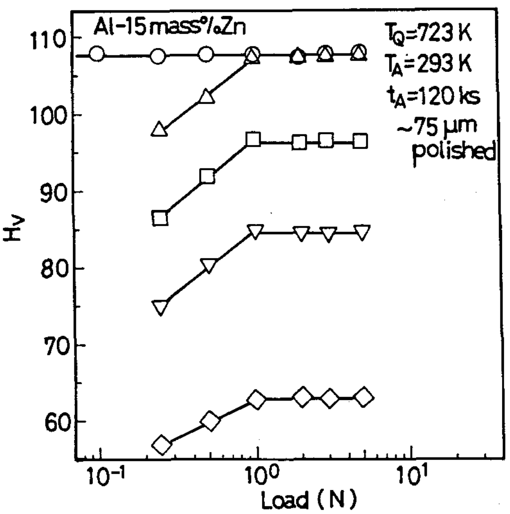


Fig.3 Dependence of hardness at position far from GB on the indentation load at several stages of isochronal annealing (marked by arrows in Fig.2).
○ 293K, △ 333K, □ 373K, ▽ 393K, ◇ 413K.

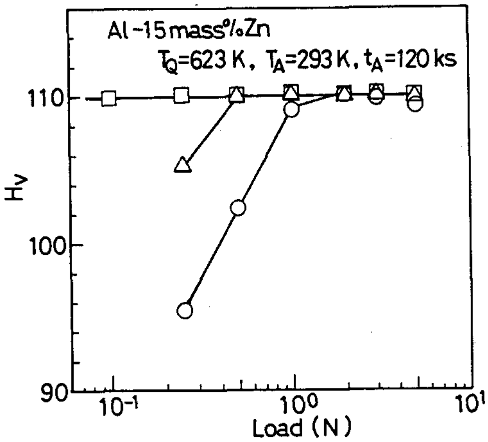


Fig.4 As Fig.1, but for the specimen aged after quenching from 623K.
○ 0μm, △ 100μm, □ 150μm.

did not occur in the interior. As the annealing temperature was elevated, hardness of the large load was also lowered, indicating the reversion in the interior, but the surface layer was softened faster at all stages up to 413K.

Fig.4 shows variation with the load, of the hardness of the alloy aged for 120ks at 293K after quenching from 623K. Lowering of quenching temperature leads to the formation of large ellipsoidal GP zones.(3) For the as-aged specimen, the hardness at 0.98N of load was lower than those at larger loads, suggesting the thicker soft layer at the surface. Removal of 100μm thickness from the surface still left soft layer, and after removal of 150μm thickness soft surface layer was completely eliminated. Isochronal annealing curves of the specimen of which the surface layer 150μm thick was removed are shown in Fig.5, classified by the position of indentation. The reversion near GB was faster and that at GB the fastest. Hardness measured at various loads was shown in Fig.6, tested at the position of indentation far from GB for the specimen isochronally annealed up to various stages of isochronal annealing. As in the above case of spherical GP zones, reversion of the large ellipsoidal GP zones began

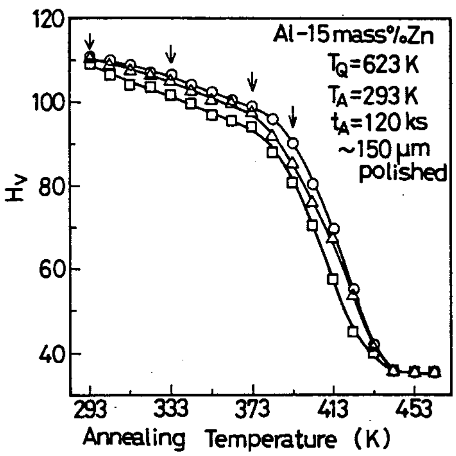


Fig.5 As Fig.2, but for the specimen aged after quenching from 623K.
○ far from GB, △near GB, □ on GB.

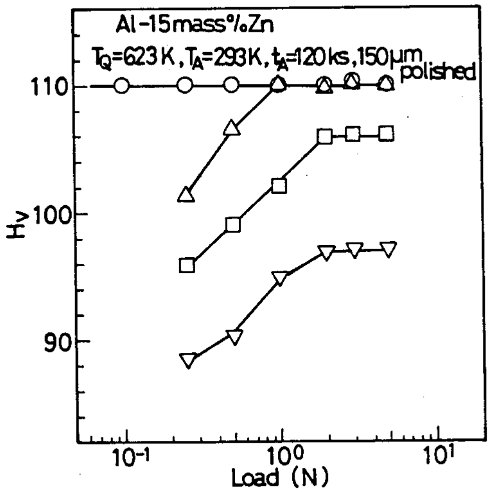


Fig.6 As Fig.3, but for the isochronal annealing of Fig.5.
○ 293K, △ 333K, □ 373K, ▽ 393K.

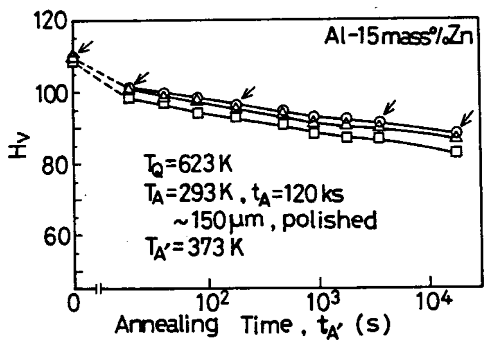


Fig.7 Isothermal annealing curves at 373K for the specimen aged after quenching from 623K and electropolished.
○ far from GB, △ near GB, □ on GB.

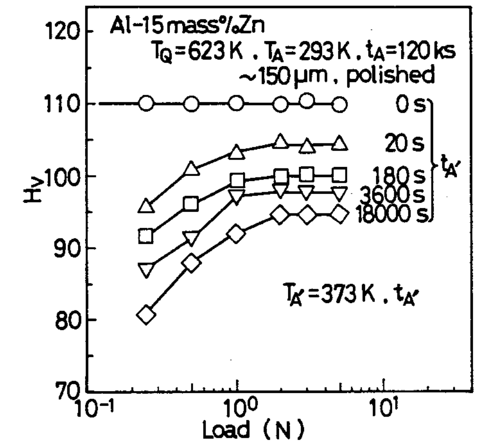


Fig.8 Dependence of hardness on the load at several stages of isothermal annealing of Fig.7. ○ 0s, △ 20s, □ 180s, ▽ 3600s, ◇ 18000s.

at the surface, and then proceeded to the interior. It is also suggested that the reversion of the ellipsoidal GP zones was slower than that of the spherical ones, perhaps due to the smaller surface-to-volume ratio. It may be considered from the thicker surface layer where reversion occurred fast than that in Fig.3 that the difference

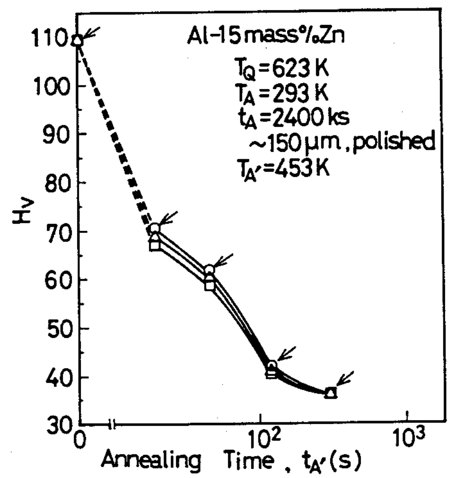


Fig.9 As Fig.7, but for the annealing temperature 453K.
○ far from GB, △ near GB, □ on GB.

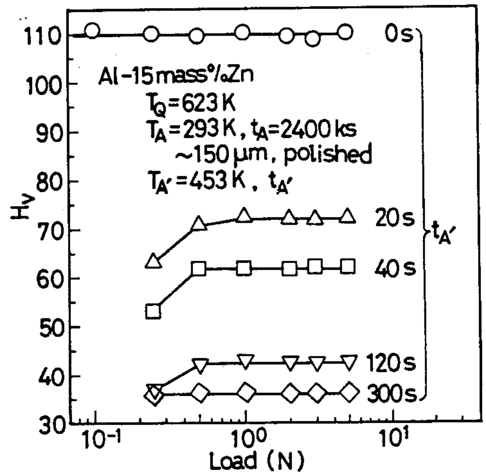


Fig.10 As Fig.8, but for the annealing temperature 453K.
○ 0s, △ 20s, □ 40s ▽ 120s, ◇ 300s.

in size of the ellipsoidal GP zones brought about larger difference in hardness than that of the spherical GP zones.

Isothermal annealing was carried out at 373K for the specimen aged for 120Ks at 293K after quenching from 623K and electropolished by 150μm. Variation of hardness with time shown in Fig.7 was depending on the position relative to GB corresponding to the isochronal experiment. At several stages of the isothermal annealing dependence of hardness number on the load of indentation was measured. The results are shown in Fig.8. The hardness in the interior decreased steadily but the hardness near the surface was lowered faster. Thickness of the layer of the fast reversion was corresponding to the load of 1.96N, which agreed well with the result of the isochronal annealing shown in Fig.6.

In the above experiment of isochronal annealing and isothermal one at 373K, partial reversion was expected to occur because of the low annealing temperature relative to the solvus of GP zones. Complete reversion at higher temperature was examined although it may be difficult to detect the dependence on the location owing to the fast development of reversion through the specimen. Fig.9 shows isothermal annealing curves at 453K for the specimen aged for 2400ks at 293K and electropolished by 150μm. Faster softening near GB was detected. Load dependence at several stages of the annealing is shown in Fig.10. Rapid reversion was observed, but it was detected that the reversion was a little faster in the surface layer which, however, was thinner than that observed in 373K annealing. The reversion was considered to be completed at 120s at the surface and at 300s in the interior.

From the results described above it is concluded that the reversion of the ellipsoidal GP zones was faster at the surface and at the grain boundary. It is considered that the surface and the grain boundary plays a role of effective source for vacancies, in addition to the interior source such as dislocations, as in the case of the reversion of spherical GP zones.

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