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Effect of Heat Treatment on the Serration of Low-Concentrated Al-Zn Alloys

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The effect of heat-treatment conditions on occurrence of serration in Al-Zn alloys was investigated. Specimens were aged for various times up to 2.6Ms at 293K or 273K after quenching from various temperatures (T_Q), 473K to 853K, and tensile-tested at room temperature. Serration occurred more easily according as T_Q became lower and the aging time became shorter: in the case that T_Q=473K serration was observed even after aging for 2.6Ms, while in the case that T_Q=773K serration did not occur irrespective of aging conditions. Serration was also recognized when the specimens were furnace-cooled from 773K to room temperature. These results together with those obtained by the electrical resistometry suggest that the serration in the low-concentrated Al-Zn alloy is caused by the formation of small GP zones whose Guinier radius is less than 1nm or some sort of solute clusters.

1. INTRODUCTION

Aging phenomena of Al-Zn alloys have been much investigated and many studies have been made on the change of mechanical properties on aging. Appearance of saw-tooth yielding (serration) in the stress-strain curve of tensile deformation has been reported in several reports. According to Yoshioka *et al.* serration was found only for low-concentrated alloys, 4%Zn or less, when aged after quenching from high temperature such as 773K. They interpreted that the appearance of serration was caused by super-jogs formed by the interaction of Frank sessile dislocation loops, which were made from quenched excess vacancies during aging, with mobile dislocations(1). This interpretation is different from that of the Portevin-Le Chatelier effect, where the saw-tooth yielding is caused by the interaction between dislocations and solute atoms(2). Later Saito *et al.* determined the parameters of discontinuous deformation, *m* and *b*, for furnace-cooled Al-10mass%Zn, considering that discontinuous deformation of this alloy was due to the interaction between dislocations and solute atoms(3). Recently Pink *et al.* suggested from the result of XSAS from Al-10mass%Zn alloy that the serration of this alloy was originated from the fomation of coherent precipitates(4).

On the other hand, it has been proposed that the serration of Al-Ag alloy, in which spherical GP zones are formed during aging as in Al-Zn alloy, is caused by cluster of solute atoms such as GP zone(5). It was reported that the serration appears when GP zones of a certain range of size were formed(6,7).

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Studies mentioned above have hardly dealt with the relation between the appearance of serration and the heat-treatment condition, especially the aging condition systematically. Detailed examination of the relation is considered to be required to reveal the mechanism of serration.

In this paper stress-strain curve of aged Al-6mass%Zn and Al-4mass%Zn alloys, where the appearance of serration has been ambiguous previously, are investigated when aged at around room temperature for various times after quenching from various temperatures and the relation between the appearance of serration and the heat treatment is discussed.

2. EXPERIMENTAL PROCEDURES

Alloys, Al-6mass%Zn and Al-4mass%Zn in nominal composition, were obtained by melting 99.996%Al and 99.999%Zn in a high alumina crucible in air. Ingots, 15mm in diameter and about 150mm in length, were homogenized for 180ks at 723K, pealed by a lathe, and forged at 723K to plates of 5mm in thickness. The plates were cold-rolled, with appropriate intermediate annealings, to strips of 0.7mm and 1.1mm in thickness. Specimens for various measurements were prepared from these strips. Shape and dimension of the specimen for each measurement were the same as reported previously[6~8].

Specimens were solutionized at 853K for 3.6ks, furnace-cooled to the quenching temperature (T_Q), 823K to 473K, kept for 3.6ks and then quenched to iced water. Aging was carried out by immersing specimens for various times in an ethanol bath kept at the aging temperature (T_A), 273K or 293K. Accuracy of the T_A measurement was within 0.5K. All specimens were equal in average grain size, about 150μm, due to the homogenization at 823K.

Specimens heat-treated were examined by tensile test with Instron testing machine, 4505, at room temperature, strain rate being $2\times10^{-4}s^{-1}$. Aging process was examined by measuring electric resistance, of which the method was described in previous reports[6~8].

3. RESULTS AND DISCUSSION

Fig.1 shows stress-strain curves of the Al-6%Zn specimen quenched from 773K and the one quenched from 473K. For the specimen T_Q=473K, serration is recognized in a wide range of strain from about 1% to 18.5%, where the stress attains the maximum value, while the specimen T_Q=773K shows a smooth change with no serration.

Fig.2 shows the collected results of the similar experiment which examined the relation of appearance of serration and 0.2% proof-stress with T_Q for the Al-6%Zn specimen. Full circles represent the case where serration was recognized. Serration was recognized in the case $T_Q \leqq$673K. According to Yoshioka *et al.* saw-tooth yielding in Al-Zn alloys was caused by the interaction of dislocations with secondary defects formed by quenching from high T_Q such as 773K. This interpretation is contradictory to the result shown in Fig.2; serration was observed in the case of low T_Q such as 523K and 473K where secondary defects should not have been formed. It was reported that secondary defects were hardly observed when the specimen was quenched from 673K or lower[1]. In Fig.2 the proof stress increases with increasing T_Q and is smaller when the serration is observed than when it is not observed. Fig.3 shows the results for Al-4%Zn specimen. The same fact is realized as for the Al-6%Zn specimen; serration appears for lower T_Q and proof stress increases with increasing T_Q.

Fig.4 shows dependence of the appearance of serration and 0.2% proof-stress on the aging time, t_A, at 293K after quenching from 473K and 573K for the Al-6%Zn specimen. Solid symbols represent the case where serration was recognized. In the case T_Q=573K the serration was observed for short t_A but it could not be found when aged for 10.8ks or longer. In the case T_Q=473K the serration was observed even after long aging such as t_A=2.6Ms. For both T_Q's, the proof stress of the case where the serration was recognized was lower than

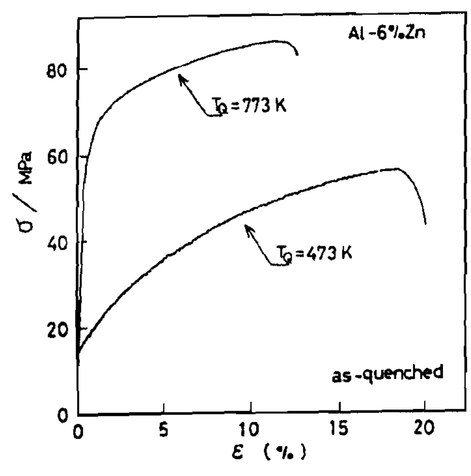

Fig.1 Stress-strain curves of the Al-6%Zn specimens water-quenched from 773K or 473K and subsequently tensile-tested.

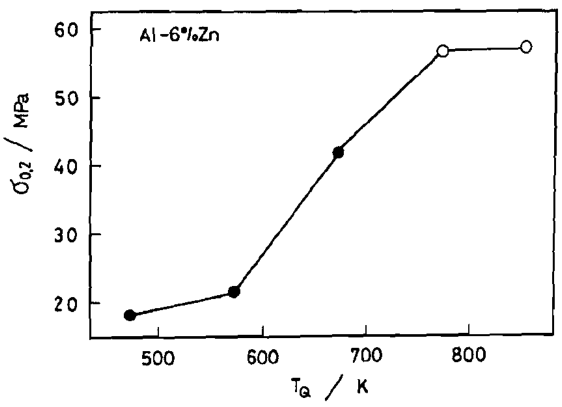

Fig.2 Effect of T_Q on the occurrence of serration and 0.2% proof stress for the as-quenched Al-6%Zn specimens. Solid and open circles represent the specimen which showed the serration and the one which did not show the serration, respectively.

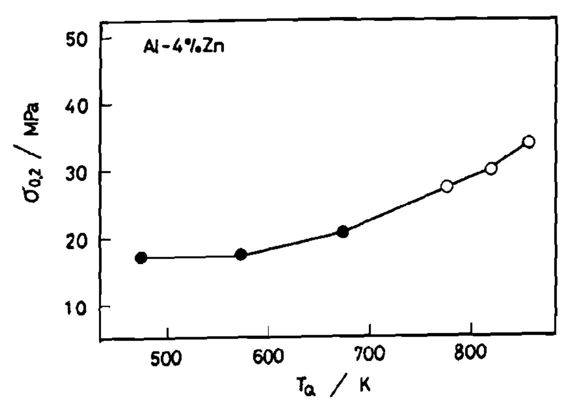

Fig.3 Effect of T_Q on the occurrence of serration and 0.2% proof stress for the as-quenched Al-4%Zn specimens. Solid and open circles represent the specimen which showed the serration and the one which did not show the serration, respectively.

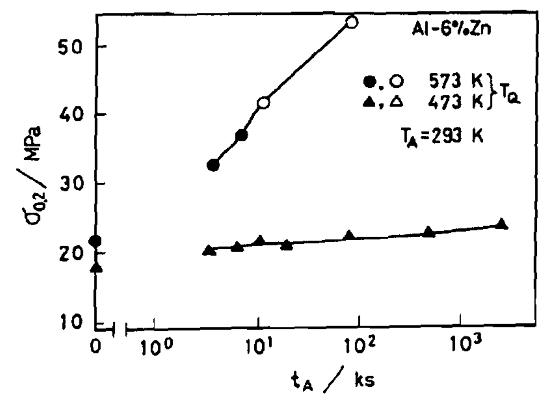

Fig.4 Effect of the aging time on the occurrence of serration and the proof-stress for the specimens aged at 293K after quenching from 573K or 473K. Solid and open circles represent the specimen which showed the serration and the one which did not show the serration, respectively.

that of the case where it was not observed. For 773K or higher T_Q (figures omitted in this paper), the serration was not observed for any t_A as was the case for the as-quenched specimen.

Fig.5 shows variation of the strain from which the serration started, ε_c, with t_A at 293K after quenching from 473K, where serration was observed for the longest t_A. The serration was retarded by longer aging. The maximum stress amplitude and the number of serration are presented as a function t_A in Figs.6 and 7, respectively. Both values remain constant for the short t_A but decrease gradually with t_A longer than 10^2ks. These results indicate that the serration became weaker according as the aging proceeded.

Fig.8 shows isothermal aging curves of resistivity when the Al-6%Zn specimen was aged at

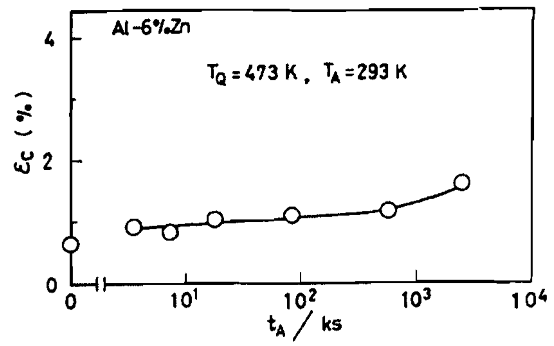

Fig.5 Relation between the strain where the serration starts and the aging time at 293K for the Al-6%Zn specimens after quenching from 473K.

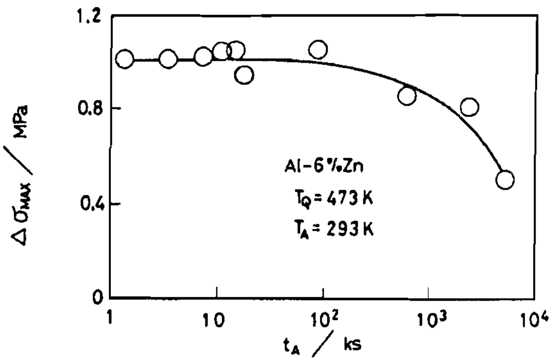

Fig.6 Variation of the maximum stress amplitude of the serration with the aging time at 293K for the Al-6%Zn specimens after quenching from 473K.

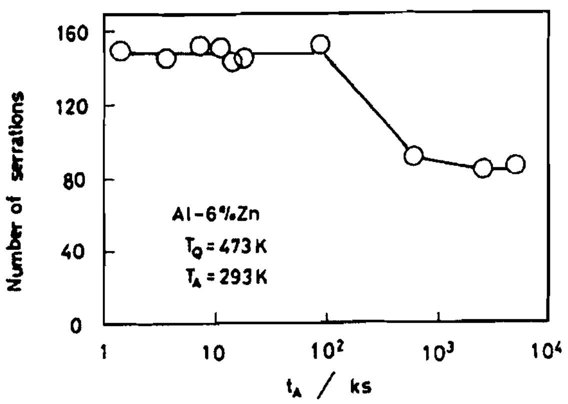

Fig.7 Variation of number of serration with the aging time at 293K for the Al-6%Zn specimens after quenching from 473K.

293K after quenching from various T_Q. Solid symbols represent the specimens which showed the serration. Curves for $T_Q \leqq 673$K show the maximum resistivity that is typical for GP zone formation. Resistivity for T_Q=773K may have taken the maximum value during quenching or within 20s of aging since the high concentration of quenched vacancy accelerated the aging rate. Isothermal aging curves at 273K showed a similar behavior. Fig.9 shows an isothermal aging curve of the Al-4%Zn specimen aged at 273K after quenching form 673K. It

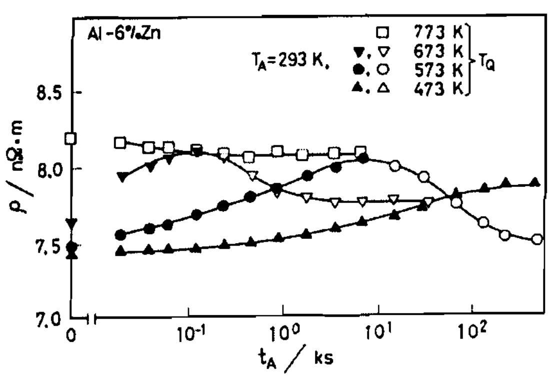

Fig.8 Variation of isothermal aging curve of Al-6%Zn specimens in electrical resistivity aged at 293K after quenching from various temperatures. Solid and open circles represent the specimen which showed the serration and the one which did not show it, respectively.

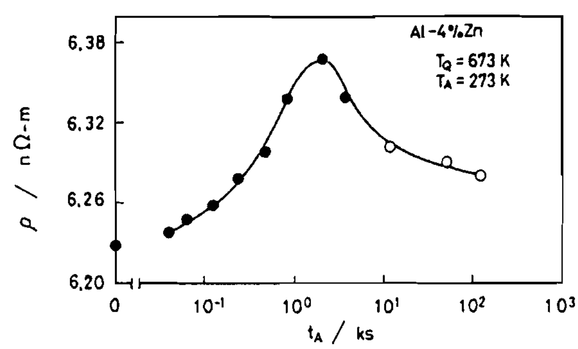

Fig.9 Variation of isothermal aging curve of Al-6%Zn specimens in electrical resistivity aged at 293K after quenching from various temperatures. Solid and open circles represent the specimen which showed the serration and the one which did not show it, respectively.

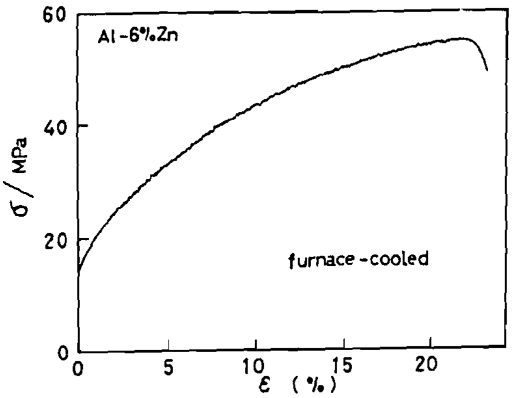

Fig.10 Stress-strain curve of the Al-6%Zn specimen furnace-cooled from 773K to room temperature.

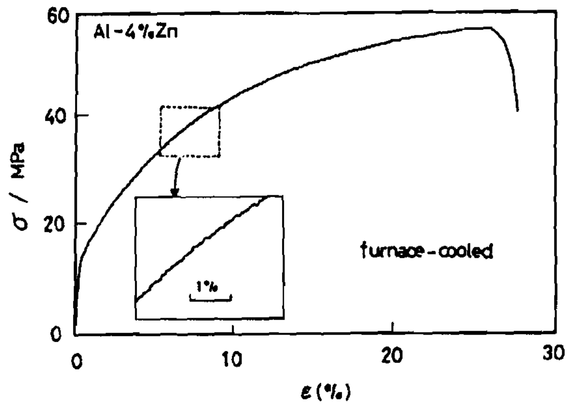

Fig.11 Stress-strain curve of the Al-4%Zn specimen furnace-cooled from 773K to room temperature.

is clearly seen from Figs. 8 and 9 that serration can be observed for the specimen aged for short time up to around the maximum resistivity. According to the studies of XSAS on the aging of Al-Zn alloy Guinier radius of GP zones formed during aging is about 1nm at the stage corresponding to the maximum resistivity(8,9). The present results might suggest that solute atoms in the matrix decreased while GP zones grew with t_A and therefore P-L effect, which was caused by the interaction of dislocations with solute atoms, became weakened. However, the specimen furnace-cooled from 773K, where solute atoms in the matrix were considered to be less than the specimen quenched from 773K, showed serration as in Figs.10 and 11, whereas the quenched specimen didn't show serration. This result is difficult to explain by the interaction between dislocation and solute atom above described.

Results of present experiment suggest a relation of the appearance of serration in Al-Zn alloy to spherical GP zones whose Guinier radius is less than 1nm or formation of some clustering of solute atoms(10,11), but it will be necessary for clear explanation of the mechanism of serration to study further the change of microstructure during deformation and the change of solute concentration during aging. Moreover, effects of test temperature and strain rate on the appearance of serration should be examined because Ikeno *et al.* (12) indicated profound effects of these factors in Al-Mg alloy.

4.CONCLUSIONS

Relation of the appearance of serration in tensile deformation to the heat treatments was examined for Al-4%Zn and Al-6%Zn alloy specimens aged at around room temperature after quenching from various temperatures between 473 and 853K.

(1) Serration was not observed for the specimens quenched from high temperatures, $T_Q \geqq 773$K.

(2) In the case $T_Q \leqq 673$K serration occurred. As T_Q became lower, the serration could be observed for the specimen aged for longer time but it was restricted in the relatively initial aging stage up to around the maximum of resistivity.

(3) Serration was recognized for the specimen furnace cooled.

(4) It is considered that serration occurs for this alloy when the specimen is heat-treated so that small spherical GP zones, whose Guinier radius is as large as 1nm, or clusters of solute atoms may be formed.

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