

## Effects of a Small Addition of Fe on Low Temperature Age-Hardening and Fatigue Strength of an Al-12mass% Zn Alloy

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**ABSTRACT** The effects of a small addition of Fe, which was often used as an additional element, on low temperature age-hardening, the formation of the soft surface layer and the microstructure of an Al-12%Zn alloy, were examined by using a Vickers microhardness tester, an Instron universal testing machine, a resistometry system, and a transmission electron microscope. Moreover, the effect of the soft surface layer on the fatigue strength was investigated by means of repeated tension fatigue test. As a result, it was found that a small addition of Fe to an Al-12%Zn alloy increased the thickness of the soft surface layer and improved the fatigue strength.

**Keywords:** *vacancy, surface, additional element, intermetallic compound, age-hardening, fatigue strength*

### 1. INTRODUCTION

Al-Zn alloy is a basic binary alloy of extra-super Duralumine. When the alloy is held at around room temperature after quenching from high temperature such as 673K, many GP zones form and contribute to age-hardening. According to a detailed examination of the age-hardening process at various regions in the specimen, at the regions near specimen surfaces and grain boundaries, the growth of GP zones is slower than in other regions[1]. The above tendency is especially remarkable at the regions near specimen surfaces. Therefore, the regions with a slightly lower hardness than inside of the specimen are formed near specimen surfaces. These regions will be called the "Soft Surface Layer". Kanadani et al. performed a fatigue test on Al-Zn alloys with various compositions and heat treatments, to investigate the effect of this soft surface layer on fatigue. They found that the existence of the soft surface layer improved fatigue strength under a repeated tensile mode[2 to 4].

By the way, it is well known that a small addition of a third element sometimes has a profound influence on the formation and growth of GP zones in Al-Zn alloys. For instance, the solvus temperature of the GP zone rises[5] and the formation of the soft surface layer is suppressed with the addition of silver[6]. Moreover it is well-known that Fe is one of the main impurities in

aluminium and forms intermetallic compounds as FeAl<sub>3</sub>[7, 8]. When the intermetallic compounds grow to the size of approximately 1 μm, they lower the toughness of Al alloys[9].

In this report, the effects of a small addition of Fe, which was often used as a small addition of a third element, on low temperature age-hardening, the formation of the soft surface layer and the microstructure of an Al-12%Zn alloy, were investigated.

## 2. MATERIALS AND METHODS

The alloys of nominal composition Al-12%Zn, Al-12%Zn-0.1%Fe, and Al-12%Zn-0.5%Fe were made from 5N aluminium, 5N zinc, and 3N iron by melting in a high alumina crucible in air. The chemical compositions of alloys were indicated in the table 1.

Alloys	Zn	Fe	Cu	Si	Al
Al-12%Zn	11.6	0.001	0.002	0.002	bal.
Al-12%Zn-0.1%Fe	11.8	0.107	0.001	0.001	bal.
Al-12%Zn-0.5%Fe	11.5	0.509	0.003	0.002	bal.

Table 1 Chemical compositions (mass%) of alloys used.

Specimens, the shape and size of which were the same as in the previous reports[10], were aged at 273 or 293K after quenching from various temperatures. These specimens were fatigue-tested under repeated tensile loading. To estimate the thickness of the soft surface layer the as-aged specimens were also examined by using a Vickers microhardness tester and an ultramicrohardness tester (MZT-1). Electrical resistivity was measured at 77K, calibrated with a dummy specimen, using a conventional potentiometric method. The microstructure of the heat-treated and fatigue-tested specimens was investigated with a transmission electron microscope (JEM-2000EX) at 200kV.

## 3. RESULTS AND DISCUSSION

### 3.1 Effect of a Small Addition of Fe on the Aging

Fig. 1 shows isothermal age-hardening curves of the binary alloy and Fe-added alloys at 293K after quenching from 673K. All curves increase initially and then arrive at a constant hardness. But as the amount of Fe increases, the hardness number becomes smaller. Also, with increasing amount of Fe, the time to attain the constant hardness becomes longer. Fig.2 shows isothermal aging curves for the tensile strength ( $\sigma_b$ ) of each alloy aged at 273K after quenching from 673K. The same effect of the addition of Fe as in Fig. 1 is noticed. Almost the same results as in Fig. 1 and 2 were obtained for the cases of quenching from 773 or 573K. According to the age-hardening isothermal aging curves for the electrical resistivity of the binary and Fe-added alloys at 273 to 473K after quenching from 623K, it is considered that a small addition of Fe does not change the solvus temperature of GP zone in the Al-12%Zn alloy [11].

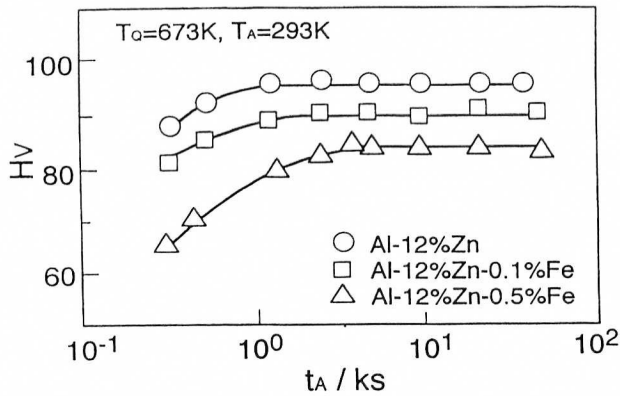


Fig.1 Age-hardening curves of the alloys aged at 293K after quenching from 673K.

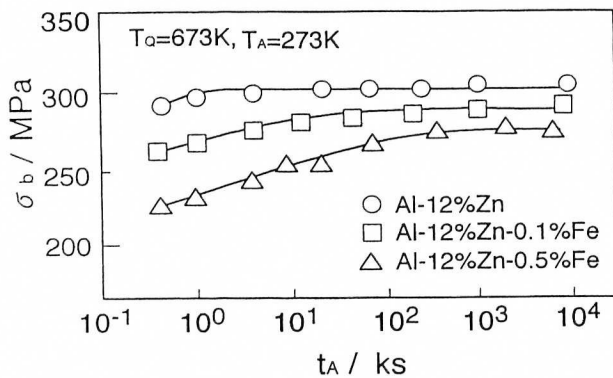


Fig.2 Isothermal aging curves for tensile strength ( $\sigma_b$ ) of the alloys aged at 273K after quenching from 673K.

Fig.3 shows the microstructures of the binary alloy and the 0.5% Fe-added alloy aged for  $6 \times 10^5$  s at 293K after quenching from 623K was investigated using a transmission electron microscope. Ellipsoidal GP zones larger than 10nm in diameter were formed in the specimen of the binary alloy, while in the specimen of the Fe-added alloy spheroidal GP zones less than 7nm in diameter were formed. Further, differences were observed in the grain size between the binary and Fe-added alloys. The size became smaller, as the amount of Fe increased.

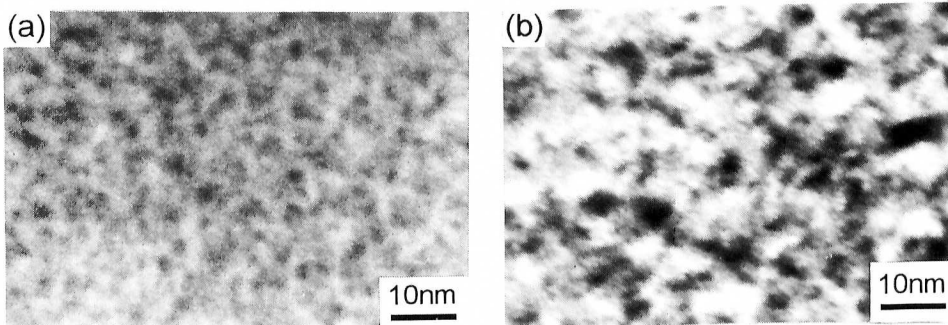


Fig.3 Transmission electron micrographs of alloys aged for 600ks at 293K after quenching from 623K. (a) Al-12%Zn-0.5%Fe (b) Al-12%Zn

In order to examine the variation of age-hardening with depth, Hv was measured at various loads from 0.01 to 9.8N. Fig.4 shows plots of hardness against load, of each alloy aged at 273K for  $1.73 \times 10^5$ s after quenching from 673K. For all alloys, the hardness is small at smaller loads, which suggests softness of the surface layer. The extent of softness becomes larger, as the amount of Fe increases. To the same purpose the surface was removed, layer by layer, by electropolishing and Hv was measured after each removal. The thickness of the soft surface layer ( $t_h$ ) evaluated in this way is  $\sim 70$ ,  $\sim 90$ , and  $\sim 120 \mu\text{m}$  for the binary, 0.1% Fe, and 0.5% Fe-added alloy, respectively. The results described above are interpreted as follows. It may be considered that the increase in the area of boundaries by grain refining and in dislocation density accompanied by the formation of metallic compounds, FeAl<sub>3</sub> (Fig. 5) which would act as sinks, results in a decrease in the concentration of quenched-in vacancies. As a result, the rate of clustering of Zn atoms is substantially reduced by the presence of Fe and then the formation of the soft surface layer is promoted.

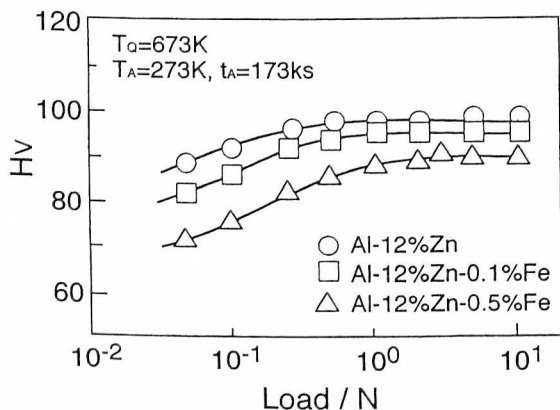


Fig.4 Dependence of Hv on the indentation load for the alloys aged for 173ks at 273K after quenching from 673K.

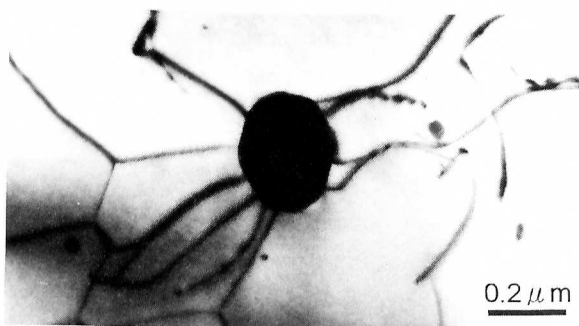


Fig.5 Transmission electron micrograph of alloy aged for 600ks at 293K after quenching from 623K. An intermetallic compound, which have grown larger than 100nm in diameter, can be seen.

### 3.2 Effect of a Small Addition of Fe on the Fatigue Strength

Fig. 6 shows the dependence of the fracture stress,  $\sigma$ , on the number of stress cycles,  $N$ , for the binary alloy and Fe-added alloys aged for  $10^5$ s at 273K after quenching from 723K. The fatigue  $\sigma/N$  curves for the Fe-added alloys are higher than for the binary alloy. As the amount of Fe increases, the curves as a whole become higher. Also, in the case of quenching from 773K the same effects of the addition of Fe as in Fig.6 were obtained.

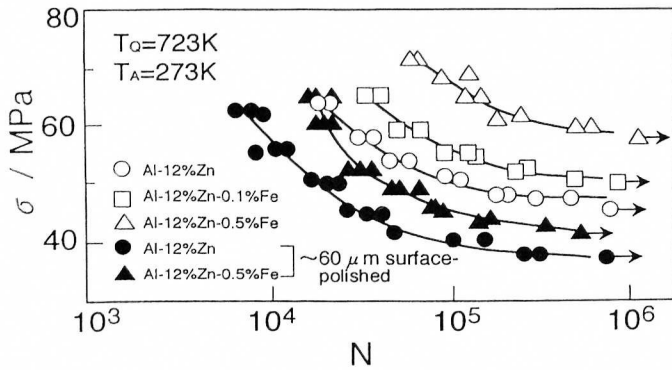


Fig.6 Dependence of the fracture stress ( $\sigma$ ) on the number of stress cycles ( $N$ ) for the specimens aged for  $10^5$ s at 273K after quenching from 723K.

From these results it is considered that a small addition of Fe to the Al-12%Zn alloy increases the fatigue strength under repeated tensile loading. The thickness of the soft surface layer for the specimen aged at 273K after quenching from 723K was  $\sim 50$ ,  $\sim 65$ , and  $\sim 90 \mu\text{m}$  for the binary, 0.1%Fe, and 0.5% Fe-added alloy, respectively. That is, the fatigue strength of the alloys containing Fe increases as the thickness of the soft surface layer increases. To investigate further the dependence of the fatigue strength of the binary and 0.5% Fe-added alloy on the soft surface layer, specimens were heat-treated in the same way as above, surface layers  $60 \mu\text{m}$  thick were removed by electropolishing and then fatigue tested. The results obtained are given in Fig.6 (●, ■). The fatigue strength of the electropolished specimen is remarkably lower than that of the as-aged specimen. Therefore, it is considered that the soft surface layer has a remarkable effect on the fatigue strength of the Al-Zn alloy containing a small amount of Fe as well as on the fatigue strength of the binary alloy; this soft surface layer has the effect of raising the fatigue strength under repeated tensile loading.

By the way, the fatigue strength of the 0.5% Fe-added alloy is higher than that of the binary alloy. This difference may be due to the strengthening of grain refining[12 to 14] and the growth of GP zones during the fatigue test[15]. According to the results of electron microscopy (Fig. 7), the average diameter of GP zones before and after the fatigue test ( $\sigma = 35\text{MPa}$ ,  $N = 10^5$ ) for the 0.5% Fe-added specimen which was aged for  $1.8 \times 10^5$ s at 293K after quenching from 623K, was 2 and 4 nm, respectively.

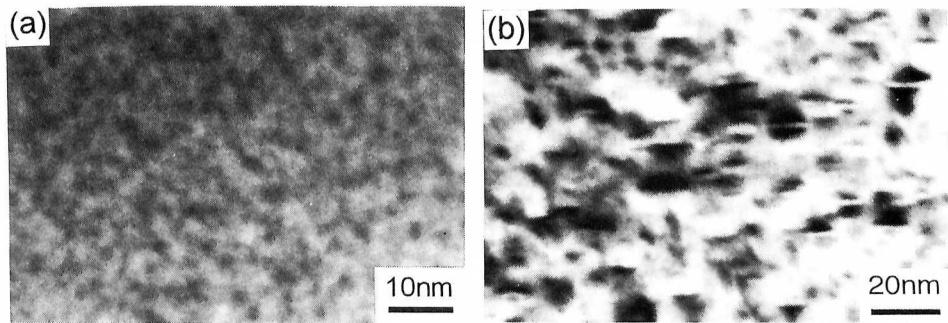


Fig.7 Transmission electron micrographs of an Al-12%Zn-0.5%Fe alloy.  
(a) before and (b) after fatigue test (stress=35MPa,  $10^4$  cycles).

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