Effect of Microstructure on Fatigue Fracture in an Aged Al-4wt%Ge Alloy

Keiyu Nakagawa¹, Teruto Kanadani¹, Norio Hosokawa¹, Tadashi Tanimoto¹, Laurence Anthony², Goroh Itoh³

¹ Department of Mechanical Systems Engineering, Okayama University of Science
² Department of Information and Computer Engineering, Okayama University of Science
³ Department of Mechanical Engineering, Ibaraki University

Keywords: Al-Ge alloy, Ge precipitates, fatigue test, precipitate-free zone, microstructure, grain boundary, aging, tensile test, vacancy

Abstract

In the paper, we report on the relationship between fatigue strength and structural changes due to aging at 473K for an AI-4%Ge alloy. Results show that the fatigue strength of the alloy decreases as the aging time increases. TEM observations of aged specimens indicate that a PFZ is formed in the vicinity of specimen surface grain boundaries, and this exhibits shear stress plastic deformation as a result of repeated loading. In addition, coarse precipitates are shown to form at specimen surface grain boundaries, and subsequently become the origin of fatigue fractures, which have a direct effect on the fatigue life of the alloy.

1. Introduction

Previous research on precipitate formation in Al-4%Ge alloys has shown that vacancy clusters are formed at the initial stage of aging, followed by the precipitation of only diamond structure equilibrium Ge phases in the matrix solid solution. In addition, it is known that vacancies play an important role during the transformation from a matrix with clusters having a coherent structure to a matrix with Ge phases having an incoherent structure [1-2]. To date, there have been many reports on the transformation of Ge phases, and it has been shown that triangular and rectangular precipitates, and bar-like precipitates are formed at the initial stage of precipitation [3-5]. Also, it has been reported that the $\{111\}_{Ge}$ plane of microscopic triangular-plate precipitates is parallel to the $\{111\}_{Al}$ plane [6-7]. On the other hand, there have been few reports on the relationship between Ge phase transformation due to aging and the fatigue strength of the alloy.

In previous work, we investigated the relationship between fatigue strength and structural changes in the vicinity of grain boundaries for Al-1.2%Si alloys under various aging conditions. It was shown that as the aging time increased, precipitates formed at the grain boundaries, and the fatigue strength decreased as the size of transgranular Si phases increased [8]. To explain the decrease in fatigue strength, we showed that dislocations accumulate in the vicinity of the crystal grain boundaries due to repeated loading, and these cause fractures to form in the vicinity of precipitates. This leads to grain boundary fractures, which subsequently causes a reduction in the fatigue strength.

In this paper, we investigate the effects of repeated loading on the fatigue strength of an AI-4%Ge alloy from the initial stage of aging to an over-aging stage.

2. Materials and Methods

Al-4%Ge alloy ingots were cast by melting 99.996% Al and 99.999% Ge in a high purity alumina crucible in air. The ingots (150 mm in length, 15 mm in diameter) were homogenized by annealing at 693K for 180ks, peeled mechanically, and then hot forged and cold rolled with intermediate annealing in air to produce 0.7 mm thick sheets. These were then used to prepare observation specimens.

Quenching was performed at a temperature of 693K (T_Q) for 3.6 ks, followed by rapid cooling in an ice water bath. After quenching, the specimens were held at 273K for 0.06 ks, followed by aging. Aging was carried out in a silicon oil bath held at 473K (T_A), at various times (t_A). The accuracy of T_A was ± 0.5 K.

For the fatigue experiments, after heat treatment, each specimen was tested on a fatigue testing machine at a stress ratio (*R*) of 0.0, a frequency of 30 Hz, and a temperature of 293 K, under various repeated tension stress amplitudes (σ), to obtain the number of repeated cycles to failure (N). For the tensile experiments, after heat treatment, each specimen was tested on an Instron testing machine at an initial strain rate of 2x10⁻⁴s⁻¹, at room temperature (293 K), to obtain stress - strain curves.

To observe the specimen structure, specimens obtained immediately after aging and specimens obtained during fatigue tests under various repeated loading cycles at $\sigma = 32.6$ MPa were electro-polished to produce thin foils. These were used for high voltage (200 kV) transmission electron microscopy (TEM) observations. In addition, in order to investigate the presence of slip bands and crack formation on the specimen surface, specimens prepared in a similarly way were observed under a metallurgical microscope. Finally, after the fatigue tests were conducted, the surfaces of specimens were investigated using a scanning electron microscope (SEM).

3. Experimental Results and Discussion

Figure 1 shows the relationship between stress amplitude (σ), and the number of cycles to failure (*N*) for samples prepared at various aging times (t_A), at a temperature of 473 K. In the figure, it can be seen that as the aging time increases (corresponding to aging progression) the fatigue strength decreases. Figure 2 shows the relationship between t_A and the tensile properties of specimens. As t_A increases, the tensile strength (σ_B) and the 0.2% proof stress ($\sigma_{a,2}$) increase, reaching maximum values at an aging time of 6 ks. At higher aging times, the tensile strength (σ_B) and the 0.2% proof stress ($\sigma_{a,2}$) decrease, suggesting that the aging has reached an over aging stage. On the other hand, the rupture elongation (*El*) shows a reverse trend.



Figure 1: Relationship between stress amplitude (σ), and number of cycles to fracture (N_t). Specimens were aged for the indicated times (t_A), at T_A = 473 K, and subjected to fatigue tests under R= 0.0.



Figure 2: Variation in proof stress, ($\sigma_{0.2}$), tensile strength (σ_B) and elongation to failure (*El*), with aging time, t_A .

In general, changes in fatigue strength correspond to changes in tensile strength, and so the decrease in fatigue strength above $t_A = 6$ ks can be easily explained. However, the results show that as the aging time increases up to $t_A = 6$ ks the fatigue strength decreases despite an increase in the tensile strength. To explain this phenomenon, we decided to investigate the transgranular precipitate structure and the precipitate structure in the vicinity of grain boundaries. Here, we describe in detail the results for secondary aging ($t_A = 0.6$ ks, UA), peak aging ($t_A = 6$ ks, PA), and over aging ($t_A = 36$ ks, OA).

Figure 3(a), (b) and (c) show TEM observations of the transgranular structure for UA, PA and OA specimens, respectively. Also shown in the figure is the average size and size distribution of plate-like Ge phases. Under all three aging conditions, the specimens show a uniform growth of Ge phases. The average size of Ge phases in the UA, PA and OA specimens were 24.5 nm, 124 nm, and 189 nm, respectively.

This suggests that increasing t_A results in the growth of Ge phases. Although the density of the Ge phases was not measured, we suggest that in the case of UA and PA specimens, the increase in σ_B and $\sigma_{a,2}$ with increasing t_A is closely related to the average dislocation migration distance in the direction of the slip planes. For UA and PA specimens,

assuming that the density remains constant, the mean free path of dislocations will be reduced as the plate-like Ge phases grow, leading to an increased fatigue strength. On the other hand, OA specimens show coarse Ge precipitates over 500 nm in size, and in the vicinity of these large precipitates, a precipitate free zone (PFZ) is observed. Therefore, the distance between Ge phases will increase greatly, thus resulting in an increase in *El*, and a decrease in σ_{B} and $\sigma_{0,2}$, as the dispersion strength becomes weaker.



Figure 3: TEM microstructure and size distribution of Ge precipitates for specimens aged at 473 K for (a) 0.6 ks, (b) 6 ks, and (c) 36 ks. *N*: total number of Ge precipitates, *n*: fractional number, *s*: mean size of Ge precipitates.

The increase in σ_B and $\sigma_{0.2}$ with increasing t_A can be explained as a result of structural changes of transgranular Ge phases. However, it is difficult to explain the decrease in the fatigue strength with increasing t_A as a result of only Ge phase structure changes. In our previous work on Al-Si alloys, we showed that structural changes in the vicinity of grain boundaries and at the specimen surface due to repeated loading had a strong influence on

the fatigue strength. In view of this, we decided to investigate if this relationship was held in the case of an AI-4%Ge alloy.

Figure 4 shows structural changes in the vicinity of grain boundaries as a result of repeated loading for a PA specimen. Figure 4(a) shows the structure before the repeated loading, and Figure 4(b) shows the structural changes resulting from repeated loading up to 30% of the fatigue life.

Following the repeated loading, the accumulation of dislocations in the PFZ can be clearly seen. Therefore, the boundary surfaces between large precipitates on the grain boundary and the matrix appear to be sites for the accumulation of dislocations.





Figure 4: TEM images in the vicinity of the grain boundaries for specimens aged for 6 ks and then fatigue tested. Number of cycles: (a) 0, (b) 1×10^5 .

Figure 5 shows the structural changes at the surface of a PA specimen as a result of repeated loading up to 30% of the fatigue life. Transgranular slip bands and slip bands in the vicinity of the grain boundary can be clearly seen, and a grain boundary crack is shown to have formed on the specimen surface (indicated by an arrow).



Figure 5: Microstructure at the specimen surface after a repeated tensile loading of 1 x 10⁵ cycles.

Figure 6(a) and (b) show the fracture surface of a PA specimen after fracture due to repeated loading. Ductile striations can be seen across the fracture surface, suggesting transgranular fracture.

Also, the formation of a fracture at the specimen surface can be observed, suggesting that the specimen surface acts as a site for excess vacancy annihilation, in a similar way to grain boundaries. From this, it can be predicted that a PFZ is formed in very close proximity to the specimen surface, and that coarse precipitates are formed on the specimen surface. In addition, at or near the intersection between the specimen surface and grain boundaries, it can be inferred that even larger coarse precipitates will be formed.

As a result, it can be predicted that at stress rates below $\sigma_{a,2}$ shear stress plastic deformation will occur preferentially at the PFZ in the vicinity of specimen surface grain

boundaries, and coarse Ge precipitates at specimen surface grain boundaries will act as sites for fatigue fractures.



Figure 6: SEM images of the fracture surface for a specimen aged for 6 ks and then fatigue tested ($N_f = 1.68 \times 10^6$). The crack indicated by an arrow in (a) is enlarged in (b).

4. Conclusions

In this paper, we have reported on the relationship between fatigue strength and structural changes due to aging at 473K for an AI-4%Ge alloy. The fatigue experiments corresponded closely with high-cycle fatigue, and the formation of fractures was shown to have a direct impact on the fatigue life of the alloy. Within the scope of the present study, it was found that shear stress plastic deformation of the PFZ in the vicinity of specimen surface grain boundaries occurred preferentially as a result of repeated loading. In addition, it was suggested that coarse precipitates that form at the specimen surface grain boundaries were the origin of fatigue fractures. From the above, we can explain the decrease in fatigue strength as the aging time increases as a result of coarse precipitation phase growth at the grain boundaries of the specimen surface.

Acknowledgments

We would like to express our gratitude to Professor Y. Yokota, Research Institute of Natural Sciences, Okayama University of Science, for assistance during the transmission electron microscope observations.

References

- [1] G.W. Lorimer: in Precipitation Processes in Solids, edited by K.C. Russel and H.I. Aaronson, AIME Conf. Proc., Warrendale, PA, 87, 1978.
- [2] E.A. Starke G. Kralik and V. Gerold, Matter. Sci. Engr., 11, 319, 1973.
- [3] S. Hinderberger, U. Dehmen, S. Xiao and H. Westmacott, Z. Metallkd., 91, 215, 2000.
- [4] S. Hinderberger, S. Xiao, H. Westmacott and U. Dahmen, Z. Metallkd., 87, 3, 1996.
- [5] U. Koster, Mater. Sci. Eng., 5, 174, 1970.
- [6] L. M. Sorokin and A. A. Sitnikova., Soviet Phys. Solid State, 9, 1525, 1968.
- [7] L. M. Sorokin and G. N. Mosina, Soviet Phys. Solid State, 10, 127, 1968.
- [8] K. Nakagawa, T Kanadani, N. Hosokawa, and T. Tanimoto, J. Japan Inst. Light Metals, 50, 650, 2000.