Article ID: 1003 - 6326(1999)04 - 0818 - 04

Suppression effect of fine Al_2O_3 particulates on aging kinetics in a 6061 matrix composite material①

Wu Gaohui(武高辉)[1,2], Ma Senlin(马森林)[1],
Zhao Yongchun(赵永春)[1], Yang Dezhuang(杨德庄)[1]
1. School of Materials Science and Engineering, Harbin Institute of Technology, Harbin 150001, P.R. China
2. State Key Laboratory of Metal Matrix Composites, Shanghai Jiao Tong University, Shanghai 200030, P.R. China

Abstract: Aging behaviors of submicron Al_2O_{3p}/6061 composite were investigated by Vickers hardness (HV) measurement and transmission electron microscope (TEM) observation. The results showed that there was an idiographic microstructure and aging characteristic in the composite. Addition of fine Al_2O_3 particulates would strongly restrain the precipitation and reduce the thermal mismatch dislocation density due to the difference of coefficient of thermal expansion (CTE) between the matrix and the reinforcement.

Key words: composite materials; particulates; aging; precipitation; vacancies **Document code**: A

1 INTRODUCTION

It has been well established that metal matrix composites(MMC) exhibited different aging kinetics compared to the unreinforced alloys because of the addition of ceramic particles or whiskers. An acceleration in the aging kinetics has been frequently reported[1~4], due to an increased dislocation density caused by the mismatch of coefficients of thermal expansion (CTE) between the reinforcement and the matrix. Meanwhile, it had also been found[5,6] that the initial stages of the precipitation sequence, i.e., Guinier-Preston (GP) zones, can be restrained. The alteration in the precipitation kinetics was taken as an attribution to a reduction in the supersaturated vacancy concentration retained in the composite vis-à-vis that of the unreinforced matrix. However, the above results of aging behaviors were normally used to applying 2 ~ 30 μm particulates as reinforcements. Thus the essential of aging kinetics of composites could not be entirely revealed. In this work, fine Al_2O_3 particualtes were used. It was found that the Al_2O_{3p}/Al composite exhibited dramatically different microstructures from the previous work, which correspondingly resulted in a good plastic deformation and high ultimate strength[7]. The effect of fine Al_2O_3 (0.4 μm) particulates on the aging behavior of the Al_2O_{3p}/6061 composite will be systematically evaluated by Vickers hardness (HV) measurements and TEM observation.

2 EXPERIMENTAL

The matrix alloy used was a 6061 aluminum alloy, whose chemical composition (mass fraction, %) is Cu 0.43, Mg 0.75, Mn 0.22, Fe 0.36, Si 1.26, Zn<0.15, Ti<0.05, and Ni<0.05, and the particulate reinforcements are fine Al_2O_3 with diameter 0.4 μm. 20% (volume fraction) Al_2O_{3p}/6061 and 30% (volume fraction) Al_2O_{3p}/6061 composites, were prepared by squeeze casting. The process of the unreinforced matrix alloy followed the same route.

① Project 59771014 supported by the National Natural Science Foundation of China
Received Oct. 26, 1998; accepted Dec. 30, 1998

The specimens used for Vickers hardness measurement, 10 mm × 10 mm × 3 mm in size, were cut by spark erosion machine from as-cast ingot. Then solution treatment was conducted in a salt bath furnace at 530 ± 2 ℃ for 1 h. After quenched in water, the specimens were aged at 145 ± 0.1 ℃, 160 ± 0.1 ℃ and 175 ± 0.1 ℃ for 0 ~ 58 h, respectively. The microstructures of the selected specimens of the unreinforced matrix alloy and composites as a function of aging time was characterized with TEM. Specimens were prepared by conventional grinding and ion milling at 5 kV and 1 mA. Bright-field electron micrographs were taken under two-beam conditions using {111} reflections.

3 RESULTS AND DISCUSSION

3.1 Hardness

Aging hardness curves of unreinforced alloy, 20% Al_2O_{3p}/6061 aged at 175 ℃ and 30% Al_2O_{3p}/6061 aged at 145, 160 and 175 ℃ are shown in Fig. 1 and Fig. 2, respectively. It is apparent that the addition of fine Al_2O_3 particulates suppressed the precipitation of the composites. For the 20% Al_2O_{3p}/6061 composite, the peak hardness occurred after 12 h rather than 5 h required by the control alloy. And the hardness of the composite changed from 145 HV to 266 HV after aged for 12 h, the aging hardening rate of the composite reached 56%, much lower than that of the control alloy (210%). However, it

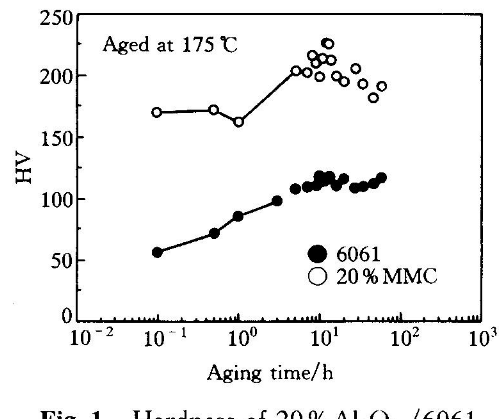

Fig.1 Hardness of 20% Al_2O_{3p}/6061 composite and its matrix aged at 175 ℃ as a function of aging time

can be found from Fig. 2 that with the increase of aging time, there is little change of age-hardening curves for 30% Al_2O_{3p}/6061 composite. This means that the precipitation of the composites can be strongly suppressed by the addition of fine Al_2O_3 particulates.

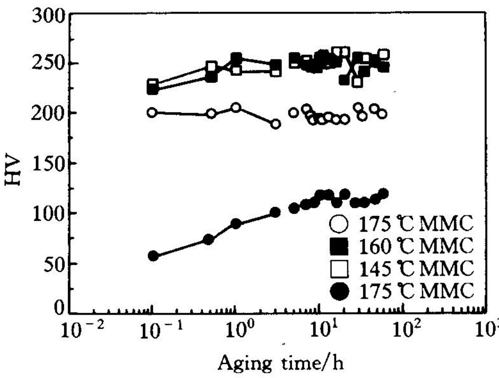

Fig.2 Aging hardness of 30% Al_2O_{3p}/6061 composite aged at 145, 160 and 175 ℃, and its matrix at 175 ℃

3.2 Microstructure

Fig. 3 shows the SEM metallographic microstructures of the test composites. It can be seen that the distribution of reinforcements is generally uniform except the 20% Al_2O_{3p}/6061 composite. In the 20% Al_2O_{3p}/6061 composite, there are particulate-rich and particulate-poor zones about 30 ~ 60 μm in diameter. This case is the result of particulate pushing effects during solidification for lower reinforcement contents. The microstructures of control alloy and composites under different aging conditions were observed by TEM. The results show that, as for the control alloy and the 20% Al_2O_3p/6061 composite the precipitates are mainly rod-like β' phases at peak age, as shown in Figs. 4(a) and (b), respectively. But the precipitates and dislocations could not obviously be observed in the 30% Al_2O_{3p}/6061 composite even aged for 58 h at 175 ℃.

Moreover, the interface between particulate and matrix was very clear. No interfacial reactants were found by TEM, as seen in Fig. 5. It is well known that the addition of particulates can give rise to high dislocation density in the matrix

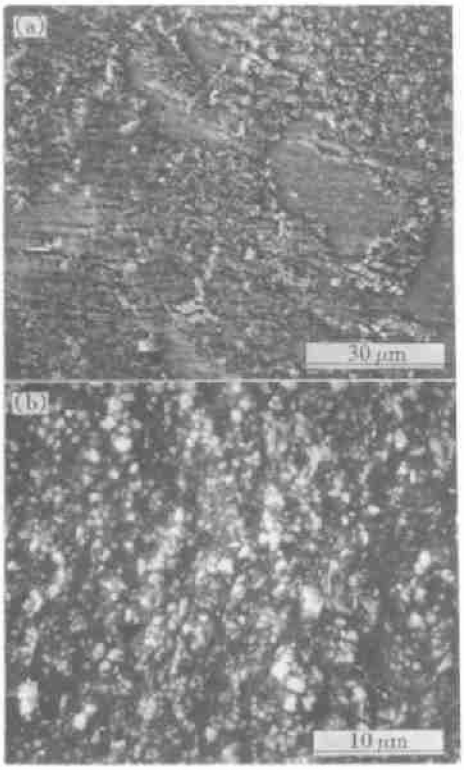

Fig.3 SEM metallographic microstructures of $20\%Al_2O_{3p}/6061$ composites
(a)—$\varphi_p = 20\%$; (b)—$\varphi_p = 30\%$

due to thermal mismatch of CTE. But this result was based on the fact that the size of particulates is more than 3 μm. When the particulate is extremely fine, the case is changed. Assuming that the particulates arrange in cube in matrix, the central distance (λ) between both particulates can be written as

$$\lambda = d/(6\varphi_p/\pi)^{1/3} \quad (1)$$

where d is the average diameter of particulates, and φ_p is the volume fraction of particulates. When $\varphi_p = 30\%$ and $d = 0.4$ μm, λ will be 0.5 μm. Then the shortest and longest distance between the surface of two particulates are 0.1 μm and 0.3 μm, respectively. After solution and quenching, thermal mismatch strain and stress would occur in the matrix. The mismatch strain (ε) can be estimated by Eq. (2) as follows:

$$\varepsilon = \Delta\varepsilon \cdot \Delta T \quad (2)$$

where $\Delta\varepsilon$ is a CTE difference between particulate and matrix; ΔT is a temperature change cooling down from solution temperature to room temperature. For the present work, the solution treatment temperature is 530 ℃, room temperature is 20 ℃, and the CTE of Al_2O_3 particulate and aluminum are 8.6×10^{-6}/K and 23.6×10^{-6}/K[10], respectively. Applying those data to Eq. (2), ε is equal to 7.65×10^{-3}. So the magnitude of mismatch strain (μ) along the axes of particulate can be given by

$$\mu = \varepsilon \cdot e/2 \quad (3)$$

where e is the shortest distance between the surface of two particulates. According to the data obtained in the present work, μ is about 0.38 nm. It could be considered that such small

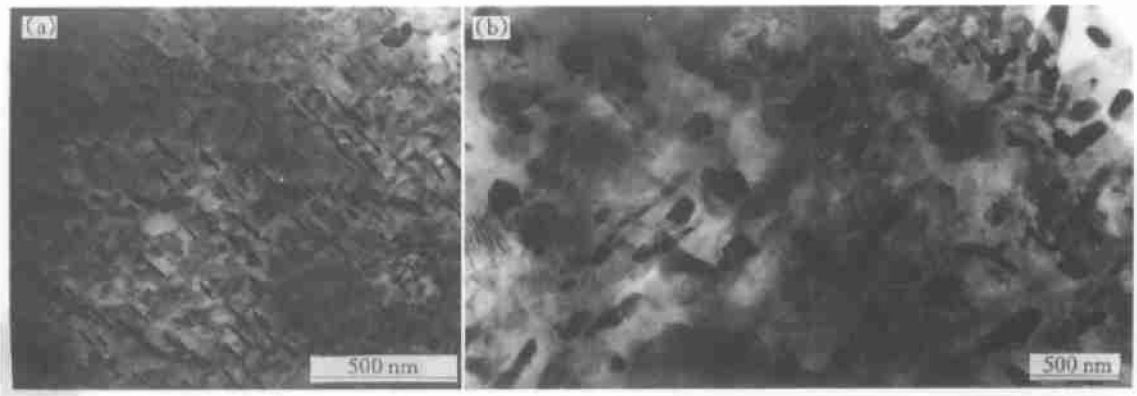

Fig.4 TEM images of β' precipitates in (a) 6061 alloy aged at 175 ℃ for 5 h and (b) $20\%Al_2O_{3p}/6061$ composite aged at 175 ℃ for 12 h (particulate-poor zones)

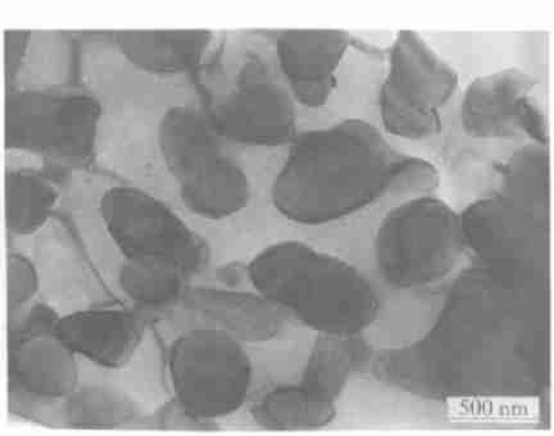

Fig.5 TEM microstructure of 30% Al_2O_{3p}/6061 composite aged at 175 ℃ for 58 h

mismatch strain could be completely depleted by lattice distortion so that the dislocation caused by thermal mismatch can not be formed in the vicinity among the fine particulates with submicron diameter.

Sannino and Rack[11], who investigated the precipitation behavior of PM2009 Al-2% SiC particulate composites, found that the larger interfacial area in the finer particulate-reinforced composites should provide an increased number of vacancy sinks. The latter would reduce the vacancy supersaturation, and then, partly suppress the nucleation of GP zones, in return, the level of precipitation hardening due to GP zones. At an equivalent volume percentage of the reinforcement, the total reinforcement/matrix surface area will increase sharply with decreasing particulate size when its size is less than 1 μm[11]. According to Salvo *et al*[12], the precipitates in unreinforced 6061 Al alloy mainly nucleate on vacancy loops. In 6061/10% SiC_p(15 μm) composites, both vacancy loops and dislocation nodes are possible. For 20% reinforcement, precipitate nucleates preferentially on dislocation nodes owing to the lack of quenched-in vacancies. But for the 30% Al_2O_{3p}/6061 composite, lower dislocation density and the lack of quenched-in vacancies result in the suppressed aging kinetics. So, it is possible to conclude that one of the most important reasons that the strong suppression of the aging precipitation of GP zones is the lack of the sites for precipitate nucleation.

4 CONCLUSIONS

(1) The precipitation of 30% Al_2O_{3p}/6061 composite is strongly restrained for the addition of submicron scale particulates. For the 20% Al_2O_{3p}/6061 composite, β' phases can precipitate in the particulate-free zones, but peak aging time is still delayed compared to 6061 Al alloy.

(2) When the reinforcement size is very fine and smaller than 1 μm in diameter, the quenching vacancies in the matrix would be absorbed by a large amount of particulate/matrix interface. And then, the vacancy saturation in the matrix becomes very low. On the other hand, the small thermal mismatch strain caused by CTE between the fine particulates and matrix would result in low thermal mismatch dislocation density.

REFERENCES

1 Janowski G M and Pleka B J. Metall Trans, 1995, 26: 3027~3035.

2 Song Y and Baker T N. Mat Sci & Tech, 1994, 10: 408~413.

3 Allen S M and Hafley J L. Metall Trans, 1991, 22A: 2553~2558.

4 Zhang Siqi, Zhang Xinming *et al*. The Chinese Journal of Nonferrous Metals, (in Chinese), 1994, 4 (4): 268~270.

5 Friend C M and Luxton S D. J Mater Sci, 1998, 23: 3173.

6 Fiorini P *et al*. Powder Metall, 1981, 4: 210.

7 Wu Gaohui, Zhao Y C *et al*. Key Eng Mat, 1995, 104~107: 467.

8 Wu Gaohui, Zhao Y C *et al*. Key Eng Mat, 1995, 104~107: 647.

9 Wu Gaohui, Kono N *et al*. Scripta Metall, 1993, 28: 683.

10 Taya M *et al*. Metal Matrix Composite. Oxford: Pergamon Press, 247.

11 Sannino A P and Rack H J. J Mater Sci, 1995, 30: 4316~4322.

12 Salavo L and Suery M. Mat Sci & Eng, 1994, A177: 19~28.

(Edited by Peng Chaoqun)