# THE 4TH INTERNATIONAL CONFERENCE ON ALUMINUM ALLOYS

# Effect of Thermal Stress on Mechanical Properties in SiCw Reinforced Al Matrix Composites

Sung-Kil HONG, Hiroyasu TEZUKA and Akihiko KAMIO Dept. of Metallurgical Engineering, Tokyo Institute of Technology, O-okayama 2-12-1, Meguro-ku,152, Tokyo, Japan

# Abstract

The relation between the mechanical properties and the thermal residual stress related to the various heat treatment processes has been studied for the unidirectionally aligned SiC whisker reinforced aluminum alloy composites. Residual stress in the matrix was measured with X-ray diffraction at triaxial stress states. The tensile residual stress was induced in the matrix of the SiCw / Al alloys composites due to the difference of the thermal expansion coefficient between the reinforcement and matrix during cooling. The tensile yield strength was decreased by the tensile residual stress, but the compressive one was increased. It became clear that the X-ray tensile residual stress on the extrusion direction in the as-quenched composites was strongly dependent on the yield strength of matrix in the composites. The tensile yield strength of the composites which restored at room temperature after cooling down to liquid nitrogen was significantly increased by the double effects due to both the decrease of tensile residual stress and the increase of the average dislocation density in the matrix of composite. In the SiCw/Al-Li-Cu composite aged isothermally after liquid nitrogen treatment, the further increase of the tensile strength was obtained compared with that of the generally aged composites.

# Introduction

In metal matrix composites, to have cooling processes from high temperatures, such as, processes of fabrication, homogenization and solutionization treatments, thermal residual stress should be induced in the composites by the difference in the coefficients of thermal expansion between the reinforcement and the matrix( $\Delta$ CTE). When the mechanical properties of the composites is investigated, in particular, the effects of thermal residual stress on them must be considered. Taya [1] has suggested that the thermal residual stress was relaxed partially by the punching of dislocations into the matrix, and so the elastic residual stress was existed in the matrix around reinforcements.

When aluminum alloy matrix composites reinforced with SiC whisker(SiCw/AI) are cooled down from high temperatures, the thermal residual stress may be induced anisotropically in the composites due to the shape of SiCw reinforcement. Most of previous studies [2], however, have not estimated the  $\sigma_{i3}$  stresses on the measurement of residual stress in the matrix measured with X-ray analysis. And also, the effects of residual stress on mechanical properties have been evaluated independently without sufficient

analysis to combine the residual stress with mechanical properties [3].

In this study, the changes of the microstructures and the elastic residual stress related to different heat treatment processes are focussed to clarify the effect of residual stress on mechanical properties in SiCw/Al composites. Furthermore, a new strengthening mechanism for SiCw/Al-Li-Cu composite achieved by a simple heat treatment method will be proposed.

# **Experimental procedure**

## Preparation of materials

Aluminum alloy matrix composites containing SiC whisker reinforcements with volume fractions from 15% to 30% were fabricated by squeeze casting method. Pure Al, Al-3mass%Mg, Al-6mass%Mg, Al-2.15mass%Li and Al-2.3mass%Li-2.8mass%Cu alloys were used as matrix alloys. Details of fabrication of the composites are represented in Ref.4. All composites were extruded with an extrusion ratio of 10:1 at 753K after homogenization for 48 hours. As-extruded Al-3mass%Mg, Al-6mass%Mg and Al-Li based alloy composites were solution

treated at 703K or 793K.

Fig.1 shows a typical microstructure of as-extruded SiCw/Al composite containing 20% Vf, where SiCw are well aligned unidirectionally along the extrusion direction. X-ray samples were cut from the center of extruded composite bars with dimensions 3mm

 $\times$  6mm  $\times$  10 mm, after treated by the various heat treatment processes. Tensile and compression test specimens were machined from asextruded composites with shapes of  $\phi$  3mm  $\times \phi$  6mm  $\times \phi$  30mm and  $\phi$  $4\text{mm} \times \phi 10$  mm, respectively, and then heat treated under the same conditions in X-ray measurement. TEM samples were prepared by cutting and mechanical polishing to a thickness of  $50\,\mu\,\mathrm{m}$ , followed by ion milling (GATAN, DUAL-ION MILL) at

Extrusion direction

 $5 \mu m$ 



4.5kV and 0.5mA. Microstructures of composites were observed by a high resolution transmission electron microscope(JEOL, 200CX) operated at 200kV.

### Heat treatment processes

In order to prevent the influence of polishing and surface oxidation, all X-ray samples were heat treated in enclosed glass tube after surface polishing with diamond paste. Heat treatment processes were four types as follows: (1) W.Q.(773K or 793K $\rightarrow$ 293K), (2) L.N.(W.Q. $\rightarrow$ 77K,1h $\rightarrow$ 293K), (3)T6(W.Q. $\rightarrow$ 473K,1h $\rightarrow$ 293K) and (4)L.N. $\rightarrow$ T6. Al-Mg alloy composites were heat treated by types of (1) and (2), and age hardenable Al-Li based alloy matrix composites were subjected with all heat treatment processes.

#### Residual stress measurements by X-ray analysis

Residual stress was measured by 331 diffraction of the aluminum matrix using  $\sin^2 \psi$  method on a X-ray diffractometer(RIGAKU, RINT2000) with Co K  $\alpha$ , operated at 40kV and 30mA. The X-ray residual stress was obtained under the triaxial stress state( $\sigma_{i3} \neq 0$ ) with the following equations proposed by Noyan [5].

$$\varepsilon_{\phi,\psi} = (s_2/2) \{ \sigma_{11} \cos^2 \phi + \sigma_{12} \sin^2 \phi + \sigma_{22} \sin^2 \phi - \sigma_{33} \} \sin^2 \psi + (s_2/2) \sigma_{33} + s_1 (\sigma_{11} + \sigma_{22} + \sigma_{33}) + (s_2/2) \{ \sigma_{13} \cos \phi + \sigma_{23} \sin \phi \} \sin^2 \psi$$
(1)

On the assumption of elastic isotropy, the X-ray elastic constants were defined,  $(s_2/2) = (1+\nu)/E$  and  $s_1 = -\nu/E$  in equation(1). In this study, the X-ray Young's moduli of the matrix alloys were assumed to be similar to that of the unreinforced matrix alloy measured by the tensile test. The values of Young's moduli for Al-Li based alloys and the other alloys were  $(s_2/2)=1.7553\times10^{-5}$ MPa<sup>1</sup>,  $1.9349\times10^{-5}$ MPa<sup>1</sup> and  $s_1=-4.395\times10^{-6}$ MPa<sup>-1</sup>,  $4.845\times10^{-6}$ MPa<sup>1</sup>, respectively. From the relation between Bragg's equation and the strain, following equations were obtained.

$$\epsilon_{\phi,\psi} = \Delta d/d_0$$
  
=(-cot  $\theta_0 \Delta 2 \theta$ )/2 ( $\Delta 2 \theta$ ; radian) (2)

$$A = \{ \varepsilon_{\phi,+\psi} \} /2,$$
  
= - (1/4) cot  $\theta_0 (2 \theta_{\phi,+\psi} + 2 \theta_{\phi,-\psi} - 2 \times 2 \theta_0) \times (\pi / 180)$  (3)

$$B = \{ \varepsilon_{\phi, \psi} \} / 2$$
  
= - (1/4) cot  $\theta_0 (2 \theta_{\phi, \psi} - 2 \theta_{\phi, \psi}) \times (\pi / 180)$  (4)

Each sample was measured at  $\phi = 0^{\circ}$ , 45° ,90° and  $\psi = 0^{\circ}$  , $\pm 16^{\circ}$  ,  $\pm 23^{\circ}$  ,  $\pm 30^{\circ}$  ,  $\pm 35^{\circ}$ ,  $\pm 40^{\circ}$ ,  $\pm 45^{\circ}$  at each fixed  $\phi$  as shown in Fig.2. Peak positions of 331 matrix diffractions were precisely determined with the center of gravity calculated using computer program and were averaged by measured at three areas. It was very difficult to obtain the lattice spacings( $d_{o}$ ) at stress free state due to the difference of the matrix composition so that the lattice spacings of as-quenched unreinforced matrix alloy were used. On the other hand, most previous studies had recognized that the residual stress of a  $\sigma_{i3}$ , was zero. Recent studies [6], however, have recognized





Fig.2 Schematic illustration of relation between extrusion direction and coordinate systems for the samples.

 $\sigma_{i3} \neq 0$ . In this work, the  $\sigma_{i3}$  stress were evaluated because Co K  $\alpha$  X-ray can penetrate approximately  $25 \mu$  m.

# **Results and discussion**

#### Microstructures

In metal matrix composites, a great deal of dislocation is generated in the matrix due to the relaxation of the thermal residual stress which was induced by the difference in the coefficient of thermal expansion during cooling. Shibata and Mori [7] showed that the average dislocation density in the matrix increased proportionally with increasing the temperature difference at cooling( $\Delta T$ ). The increase of the average dislocation density in the matrix give rise to the increase in yield strength( $\Delta \sigma_{ey}$ ) of composites as shown by the following equation(5).

$$\Delta \sigma_{\mu\nu} \rightleftharpoons \alpha \,\mu \,\mathrm{b} \,\rho^{\,\nu 2} \tag{5}$$

where b is Burgers vector,  $\rho$  is the increase in dislocation density over that of the matrix density and  $\alpha$  is a geometric constant. Hansen [8] obtained a value for  $\alpha$  of 1.25 for aluminum. As shown in Fig.3, the dislocation density in the L.N. composite is higher than the W.Q., as expected. If the dislocations in the L.N. composite didn't poligonize during heating up to room temperature, the average dislocation density of the L.N. composites will be much higher than that of the W.Q. composites. It can be expected the yield strength of the L.N. composites will further increase due to the increase of dislocation density. Because the temperature difference( $\Delta$ T) in the L.N. composites was a much larger than that in the W.Q. composites.

#### X-ray residual stress

The residual stresses caused by W.Q. and L.N. treatments in SiCw/PureAl composites containing whisker of 20% and 30% are shown in Fig.4. Principal stress components ( $\sigma_i$ ) indicate a high tensile residual stress, while the shear residual stress( $\sigma_{ij}$ ) shows a very small value. The whisker axial stress( $\sigma_{11}$ ) in the W.Q. composites indicates the largest value, and those of the perpendicular directions to the extrusion direction are approximately same values. The  $\sigma_{ii}$  of the W.Q. composite increases with increasing whisker Vf. In all composites, in particular, the tensile residual stresses( $\sigma_{ii}$ ) extensively decrease by L.N. treatment compared to them of the W.Q. composites. The reason why the above result that the compressive stress is induced by heating from liquid nitrogen temperature to R.T..

### Relation between residual stresses and yield strengths

The relation between tensile yield strengths of 0.02% and 0.2% off sets and X-ray residual stress( $\sigma_{11}$ ) in the water-quenched SiCw/Al alloys composites are shown in Fig.5. The tensile yield strengths increase with increasing the yield strength of the matrix in the composite, simultaneously, the values of  $\sigma_{11}$  tend to increase with increasing the tensile yield strengths in the composites. In almost all of the composites, the values of  $\sigma_{11}$  are good agreement with the values between 0.02% and 0.2% off sets in the various

SiCw/Al alloys composites. The above result can be applied to estimate the tensile yield strengths of the water-quenched SiCw/Al composites without the tensile test.

Table 1 shows the difference ( $\Delta \sigma_{0,2} = \sigma^{\text{Com}_{-}} \sigma^{\text{Tens.}}$ ) of 0.2% yield strength in the tensile test and that in the compressive test for variously heat treated composites containing 20% Vf. The  $\Delta \sigma_{0,2}$  of the W.Q. and the L.N. composites becomes larger with increasing magnesium content, which leads to the increase of matrix yield strength in the composites.



500nm



500 n m

Fig.3 TEM images of the SiCw/Al-6mass%Mg composites (Vf=20%), which are (a) W.Q. and (b) L.N. treated composites. Dislocation density in the matrix of the L.N. composite is much higher than that of the W.Q. composite.



Fig.4 The measured residual stresses in the W.Q. and the L.N. treatment specimens for the SiCw/Pure AI composites containing whisker Vf of (a) 20% and (b) 30%.



Fig.5 Relation between the 0.02% and 0.2% off sets in the tensile strength and the residual stress of the whisker axial direction. The whisker axial stresses agree well with the values between 0.02% and 0.2% off sets in the tensile one.

The  $\Delta \sigma_{02}$  of the L.N. composites is much larger than that of the W.Q. composites with the exception of the Al-Li binary alloy matrix composite. Because the phase transformation takes place during cooling from the solution treatment temperature in Al-Li alloy, the influence of residual stress and dislocation on the yield strength in Al-Li alloys appears to be different from the other aluminum alloys. Arsenault et al. [10] suggested that the existence of the tensile elastic residual stress in the matrix induced during cooling decreases the tensile yield strength in the SiC/Al alloys composites, but it increases the compressive one. Thus, the  $\Delta \sigma_{02}$  consequently indicates a positive value. This is well agreed with the results obtained in this work.

To clarify the negative effects of tensile residual stress generated by various heat treatments on the tensile yield strength of the composites, the calculated reduction ratios(e) of

Matrix alloys (mass%)	W.Q. (MPa)			L.N.(MPa)		
	$\sigma^{\text{Comp.}}_{0.2\%}$	σ <sub>0.2%</sub> <sup>Tens.</sup>	c(%)	$\sigma_{0.2\%}^{\text{Comp.}}$	$\sigma_{0.2\%}^{\text{Tens.}}$	c(%)
Pure A1	206.7	156.5	15.8	186.5	165.9	6.2
Al-3Mg	338.7	239.0	20.9	346.7	295.6	8.6
Al-6Mg	436.5	312.0	20.0	457.1	366.7	12.3
Al-2.15Li	269.9	220.0	11.1	291.0	238.0	11.1
Al-2.3Li -2.8Cu	378.0	231.0	31.8	418.0	356.0	8.7

Table 1 The reduction ratios(e) of the tensile residual stress on the tensile yield strength of the SiCw/A1 alloys composites containing 20% Vf.

\* Note: reduction ratio(c) = {(  $\sigma_{0.2\%}^{\text{Comp.}} - \sigma_{0.2\%}^{\text{Tens.}}$ ) / 2 } /  $\sigma_{0.2\%}^{\text{Tens.}} \times 100(\%)$ 

the W.Q. and the L.N. composites containing whisker Vf of 20% are shown in Table 1. The reduction ratio(e) was calculated as follows:  $c(\%) = (\Delta \sigma_{0,2}/2) / \sigma^{\text{Tens.}} \times 100$ . The average reduction ratio in the L.N. composites is approximately 10% and is smaller than that of the W.Q. composites which is 20%. The decrease of the reduction ratio by L.N. treatment resulted in the reduction of tensile residual stress in the matrix during heating up to R.T., and then the increase of the tensile yield strength is obtained in the L.N. composite.

The important point to notice in the L.N. treatment is that the tensile yield strength is significantly increased by the double effects which are both the reduction of tensile residual stress and the increase of average dislocation density in the matrix.



Fig.6 Relation between the tensile yield strengths and the various heat treatments on the SiCw/Al-Li-Cu alloy composites containing whisker Vf of (a) 15% and (b) 20%. The tensile yield strength in the L.N. - 473K,1h composite shows the largest value.

# Application of L.N. treatment

In our previous study [11], it was clarified that a remarkable age hardening in SiCw/Al-2.3%Li-2.8%Cu alloy composite was obtained by the precipitation of T1(Al\_CuLi) phase which precipitated preferentially on the dislocation. As mentioned before, the dislocation density in the L.N. composite is much higher than in the W.Q. one. The combination of the L.N. treatment and age hardening treatment was applied to the SiCw /Al-Li-Cu alloy composites. Fig.6 shows the tensile yield strengths(0.2% off set) of the SiCw / Al-Li-Cu alloy composites containing 15% and 20% Vf. In all composites, the composites T6 treated after L.N. treatment show the largest tensile yield strength. This result can be explained by combining the strengthening mechanism of L.N. treatment with the age hardening.

## Conclusions

(1) The tensile residual stress in the unidirectionally aligned SiCw/Al alloy matrix composites is induced anisotropically during cooling from solutionization temperature. In the W.Q. composites, the stress( $\sigma_{11}$ ) in the whisker axial direction is the largest one, the  $\sigma_{22}$  and  $\sigma_{33}$  were approximately same ( $\sigma_{22} = \sigma_{33}$ ) and the  $\sigma_{11}$  agrees with the value between 0.02% and 0.2% off sets of tensile strength.

The tensile residual stress in the L.N. composites is reduced by heating from liquid nitrogen temperature to R.T.. The largest reduction of tensile residual stress caused by L.N. treatment is obtained in the  $\sigma_{ij}$  stress.

(2) The negative effect of the tensile residual stress on the tensile yield strength of unidirectional SiCw/Al alloy matrix composites becomes larger in the W.Q. composites than in the L.N. composites.

(3) The tensile yield strength of the L.N. composites is sufficiently increased by the double effects which are the reduction of tensile residual stress and the increase of dislocation density.

(4) By combining the L.N. treatment effect with the age hardening, the tensile yield strength in the SiCw/Al-Li-Cu alloy composites is increased much more compared to that of the generally T6 treated composites.

# References

- 1. M.Taya: The Japan Institute of Light Metals, 42(1992), 524
- 2. C.S.Barrett and T.B.Massalski:Struc. of Metals,(1966),466
- 3. Z.M.Sun, J.B.Li, Z.G. Wang and W. J.Li: Acta metall., 40(1992), 2961
- 4. S.K. Hong, H.Tezuka and A.Kamio: The Japan Institute of Light Metals, 43(1993), 82
- 5. I.C.Noyan:Metal. Trans.,14A(1983),249
- 6. H.M.Ledbetter and M.W.Austin:Mater. Sci. and Engi., 89(1987), 53
- 7. S.Shibata and M.Taya, T.Mori and T.Mura: Acta metall. mater., 40(1992)
- 8. N.Hansen:Acta metall.25(1977),863
- 9. R.J.Arsenault and M.Taya:Acta metall.,35(1987),651
- 10, S.K. Hong, H.Tczuka and A.Kamio: TheJapan Institute of Light Metals, 43(1993), 328

# THE 4TH INTERNATIONAL CONFERENCE ON ALUMINUM ALLOYS

## FRACTURE BEHAVIOR OF LAMINATED DISCONTINUOUSLY REINFORCED ALUMINUM MATERIAL

T. M. Osman<sup>1,2</sup>, J. J. Lewandowski<sup>1</sup>, D. R. Lesuer<sup>2</sup>, C. K. Syn<sup>2</sup>, and W. H. Hunt, Jr.<sup>3</sup>

Dept. of Materials Science and Engineering, The Case School of Engineering, 1. Case Western Reserve University, Cleveland, OH 44106, USA 2. Materials and Manufacturing Engineering Section, Lawrence Livermore National Laboratory, Livermore, CA 94551, USA 3.

Alloy Technology Division, Alcoa Technical Center, Alcoa Center, PA 15069, USA

## Abstract

Laminated metallic composites are being developed for applications which require high specific stiffness and fracture resistance. Recent work with laminated discontinuously reinforced aluminum (DRA) materials has demonstrated the potential for marked improvements in stable crack growth resistance via extrinsic toughening. The purpose of this work is to compare the fracture mechanisms and fracture resistance of laminated DRA materials to unlaminated DRA materials. In particular, the production of extensive stable crack growth and the associated improvement in damage tolerance in DRA laminates is documented.

## Introduction

Discontinuously reinforced aluminum (DRA) materials benefit from an enhanced specific stiffness which scales directly with the reinforcement volume fraction. This increase in modulus, however, is accompanied by a decrease in fracture resistance when compared to the unreinforced matrix.[1-4] DRA plate and sheet product exhibits a macroscopically brittle fracture where an approximately flat fracture surface is produced. Fracture often occurs with little, if any evidence of stable crack growth. Even in cases where some stable crack growth is observed, the degree of crack stability is much less than that for an unreinforced aluminum alloy of similar composition. [2,4,6,7] While the fracture resistance of DRA materials may be improved via manipulations in particle size, particulate volume fraction, matrix composition, thermal treatment, and specimen thickness [1-6], further improvements are necessary for more widespread structural application.

As a result, much research has focused on efforts to improve the fracture toughness of DRA materials.[2-9] The extent of possible improvement in intrinsic fracture resistance appears to be limited by the inherent damage mechanisms which operate in these materials.[5] For this reason, extrinsic toughening mechanisms have been proposed as a route for the improved fracture resistance of DRA materials.[2,5,8-18] As reviewed elsewhere[18], the goal of extrinsic toughening is to reduce the driving force for crack propagation by changing the stress state at the crack tip.

One method of utilizing extrinsic methods for toughness improvements on a macroscopic scale in DRA materials is through laminated structures. [2,9-14,15-18] Previous work has been performed on laminates consisting of layers of monolithic metals [20-24], but recent efforts have focused on laminated structures containing alternating layers of DRA materials and unreinforced