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**PROCESSING, MICROSTRUCTURE AND PROPERTIES
OF AL-SiC COMPOSITES
PRODUCED BY POWDER METALLURGY**

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ABSTRACT

A wet blending process and a sol gel process for preparing powder metallurgy Al-SiC composites have been described. The improvement in microstructures and mechanical properties of the composites as results of these processes, specially the enhancement of thermal stability as a result of sol gel treatment of SiC, have been demonstrated. The discontinuous Al-SiC composites can be thermomechanically processed using the technologies developed for conventional materials. The microstructure and texture development of Al-SiC composites during cold rolling and heat treatment has therefore been studied and discussed.

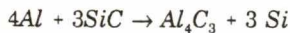
1. INTRODUCTION

Metal matrix composites (MMCs) are well established materials which have been on the market since the early 1980s. The Al-SiC MMCs are of great interest, especially in the automotive and aerospace industries, due to their high specific strength.

The Al matrix can be reinforced by continuous SiC fibres or short SiC fibres, whiskers, or particulates which have much higher strength and elastic modulus than the matrix. The Al-SiC MMCs can be produced by powder metallurgy or molten metal routes, such as squeeze casting or co-spraying. For discontinuous Al-SiC, the billets and ingots can be re-shaped using traditional technologies such as extrusion, rolling and forging. The as-supplied composite ingots can also be remelted and recast into a near net-shape.

The development of Al-SiC MMCs has to cope with a number of difficulties, such as:

- (1) uniformly distribute the SiC reinforcement in the matrix.
- (2) reduce the breakage of the SiC reinforcement, particularly SiC whiskers, during fabrication.
- (3) limit chemical reactions between SiC reinforcement and molten Al, which degrade the reinforcement. The main reaction between SiC and molten Al is:



- (4) control the product quality during thermomechanical processing.
- (5) improve the mechanical properties through improvements of the above.

The present paper will concentrate on Al-SiC composites produced by powder metallurgy (PM) technique. A wet blending and a sol gel process for preparing PM Al-SiC composites has been developed. The improvement in microstructures and mechanical properties as results of these processes, especially the enhancement of thermal stability as a result of sol gel treatment of SiC, have been established. Additionally the microstructure and textural development of Al-SiC composites during cold rolling and heat treatment has been studied and discussed.

2. MATERIALS AND PROCESSING

Characteristic parameters for atomized Al powder supplied by Metals Disintegrating, USA, SiC whiskers (SiC_w) supplied by Tokai Carbon Co., Japan, and SiC particles (SiC_p) supplied by Elektroschmelzwerk Kempten, Germany are listed in Table 1. About 1 wt% Al₂O₃ present in the Al is originating from the surface oxide on the Al powder.

The manufacturing process consists of dry blending of Al powder and SiC in a Waring Blender, cold compaction, degassing for 6 h at 450°C in vacuum, hot compaction at 550°C and extrusion. The extrusion ratio is 15:1 and the extrusion temperature is 500°C.

Table 1. Raw materials

	Diameter (μm)	Length (μm)	Al ₂ O ₃ (wt%)	Fe (wt%)	Si(wt%)
Al powder	6.4	-	1.0	0.26	0.18
SiC whisker	0.1-1.0	30-100	-	-	-
SiC particle	0.8 3 12 17	-	-	-	-

3. ULTRASONIC WET BLENDING

The Tokai SiC_w used in the present work have a strong tendency to form nests of entangled material. Fig. 1(a) shows an example of the as-received SiC_w. The standard dry blending cannot deagglomerate the SiC_w nests effectively. About 50% by volume of SiC_w remain in agglomerates approximately 100 μm in diameter in extruded composites as shown in Fig. 1(b). Additionally, during standard dry blending the SiC_w are severely damaged, decreasing

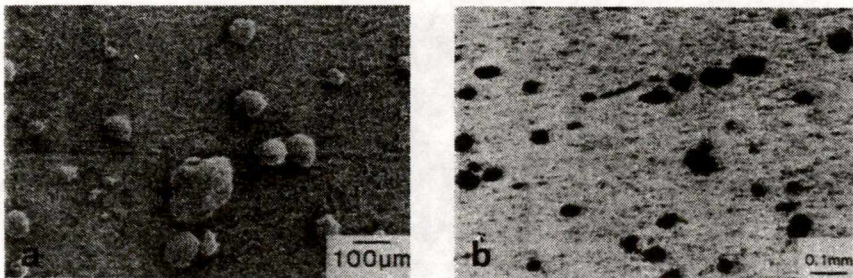


Fig. 1 (a) SEM micrograph of as-received SiC whiskers.
(b) Optical micrograph of an extruded Al-10 vol%SiC_w prepared by standard dry blending. The black inclusions approximately 100 μm in diameter are SiC_w agglomerates.

the whisker length by more than 50%. The agglomeration and breakage of SiC_w reduce their effectiveness as a reinforcement.

These problems can be limited by replacing the dry blending with an ultrasonic wet blending. Using a Branson ultrasonic generator and transmitting crystal, high frequency ultrasonic energy is transmitted through a titanium horn into an isopropylalcohol solution. When the SiC_w are added to the isopropanol, the nests are broken up and SiC_w separated. The mixture is kept cool by placing the mixing container in a water/liquid nitrogen bath. The Al powder is slowly added to the SiC_w-isopropanol mixture and complete dispersion and mixing is obtained after one hour. The blended Al-SiC_w is then separated from the isopropanol by filtering and then dried under vacuum at 80°C for 14 hours. The cold, hot compaction and extrusion follow standard routines mentioned in section 2.

Microstructure and properties

Fig. 2 is a micrograph showing an extruded Al-10 vol% SiC_w prepared by ultrasonic mixing. The improved SiC_w distribution and uniformity of this material is evident compared to Fig. 1(b). In addition, the whisker damage is reduced. Table 2 gives the length measurements of the SiC_w extracted from the extruded Al-10 vol% SiC_w prepared by wet and dry blending respectively. The resulting whisker aspect ratio is nearly doubled following the wet blending process.

Table 2. SiC_w Length after extrusion

Blending method	Length (μm)	Aspect ratio*
Dry	2.8 ± 2.3	5.5
Wet	4.5 ± 3.0	9.0

SiC_w diameter = 0.5 μm

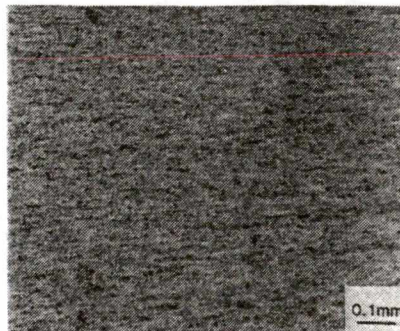


Fig. 2 Optical micrograph of an extruded Al-10 vol%SiC_w prepared by ultrasonic wet blending.

Table 3. Tensile properties of Al-10 vol% SiC_w

Blending method	Elastic modulus E (GPa)	Yield Strength $\sigma_{0.2}$ (MPa)	Ultimate strength σ_{UTS} (MPa)	Strain to failure ϵ (%)
Dry	77	111.6 \pm 2.0	227.6 \pm 1.0	17.0 \pm 0.7
Wet	80	137.9	264.5	12.7

Table 3 shows that the wet blended Al-SiC_w has higher strength and lower ductility than the dry blended materials. The strength increase for the wet blended Al-SiC_w can be attributed to a more homogeneous microstructure, a better alignment of SiC_w and to an increased whisker length. With the elimination of the whisker agglomerates a larger volume fraction of the SiC_w is able to effectively reinforce the composites. As the whisker length increases, SiC_w bear higher stress when the material is loaded. This effect increases the strength of the composite, but may lead to more SiC_w fracture and reduces the strain to failure.

4. SOL GEL TREATMENT

A process has been developed to produce Al-SiC composites in which the SiC_w or SiC_p are coated with alumina using a sol gel technique. The alumina sol gel treatment produces a coating layer on the SiC which promotes uniform distribution (in the case of SiC_w) and reduces thermal reaction with the Al matrix (in the case of both SiC_w and SiC_p).

An aqueous sol with a boehmite content of 2 to 10% by weight is prepared on the basis of a commercial boehmite (AlOOH) supplied by Condea Chemie of Hamburg, Germany. The coating process involves adding the SiC to the sol and stirring, followed by drying at 40°C in vacuum and at 450°C in air [1]. This process has been protected by a patent pending [2]. During the sol gel treatment the boehmite particles (5 - 10 nm size) of the sol are adsorbed onto the surface of the SiC to form a coating layer. The thickness of the coating layer is controlled by the boehmite concentration of the sol and the SiC concentration added in the sol. The nominal thickness can be calculated, and this value will be used in this text to define the coated SiC type, e.g. SiC (40 nm) means SiC coated with a 40 nm layer. The treated SiC is then mixed with Al powder by means of dry blending followed by standard routines.

Microstructure and properties

The deposition of boehmite particles on SiC_w causes SiC_w deagglomeration and uniform dispersion in the aqueous suspension. The uniformity is maintained when the coated and dried SiC_w are blended with the Al powder and compacted. As a result, the composites containing coated SiC_w has a more homogeneous microstructure and the homogeneity increases with increasing coating thickness. When the coating thickness is 40 nm, the Al-SiC_w composite is free from whisker agglomerates as shown in Fig. 3. TEM examination shows that in the composite containing coated SiC_w (40 nm), the alumina coating on SiC_w varies from 20 to 100 nm and all the whiskers are coated. An example is shown in Fig. 4. The Al matrix in the

composites contains a fine dispersion of alumina particles originating from the surface oxide on the Al powder. The composites with coated SiC_w additionally contain alumina from the sol, a part of which does not adhere strongly enough to the SiC_w surface and is dispersed into the matrix during processing.

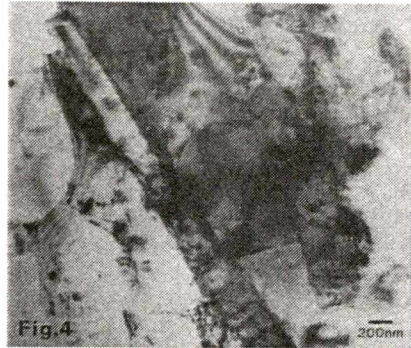
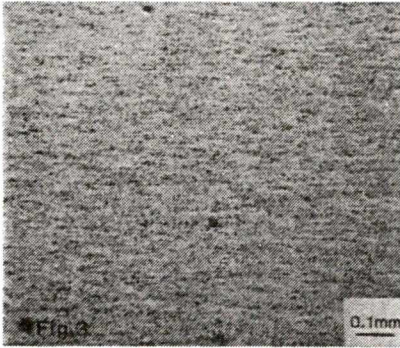


Fig. 3 Optical micrograph of an extruded Al-10 vol%SiC_w in which SiC_w are sol gel coated (40nm).

Fig. 4 TEM micrograph showing a coated (40nm) SiC whisker.

Table 4. Mechanical properties

	E (GPa)	$\sigma_{0.2}$ (MPa)	σ_{UTS} (MPa)	ϵ (%)
Al-10 vol% SiC _w (uncoated)	77.2	111.6	227.6	17.0
Al-10 vol% SiC _w (coated, 15 nm)	86.5	127.6	278.8	16.5
Al-10 vol% SiC _w (coated, 40 nm)	82.0	134.0	293.4	18.6
Al-20 vol% SiC _w (uncoated)	84.7	139.1	220.5	3.1
Al-20 vol% SiC _w (coated, 15 nm)	92.7	188.0	308.0	3.8
Al-10 vol% SiC _p (12 μ m) (uncoated)	90.0	117.6	204.8	16.3
Al-10 vol% SiC _p (12 μ m) (coated, 40 nm)	90.0	122.9	206.3	14.1

The E modulus and tensile strengths of composites containing coated SiC_w increase significantly in comparison to the composite with uncoated SiC_w as shown in Table 4. The increase of E modulus and strength is mainly due to a more homogeneous distribution and a better alignment of SiC_w , which ensures a more effective usage of the reinforcement, and the increased concentration of dispersed alumina fine particles in the matrix.

Table 4 also includes the data for Al- SiC_p composites. No significant increase in mechanical properties has been found in Al- SiC_p composites as a result of sol gel treatment. This is mainly because SiC_p do not form nests. The distribution of SiC_p in the composites is reasonably uniform even without sol gel treatment.

Thermal stability

A reaction between molten Al and SiC takes place in composites prepared by the liquid metal route forming Si and Al_4C_3 [3] which degrades the reinforcement. To avoid this reaction, research has been directed towards the design of thermal stability - enhancing procedures. These procedures consist either in adding alloy elements to the metal [4], or in depositing a suitable layer on the surface of the reinforcing bodies [5]. The experimental evidences show that the present sol gel treatment produces an interphase layer on the SiC and reduces thermal reaction with the molten Al. Tests are carried out to determine the extent of the reaction on as-received SiC and on the same SiC coated according to the present process. The tests consist of remelting Al-10 vol% SiC composites (prepared by the powder route) in a vacuum ($<10^{-5}$ mm Hg) or in Ar, cooling down to room temperature followed by DTA (Differential Thermal Analysis) and XRD (X-Ray Diffraction) analysis to determine the concentration of Si dissolved in the Al matrix as a consequence of the reaction at high temperature. The temperature of 730 and 750°C for various time lengths are chosen as normal for the molten metal route operations in practice.

Typical DTA curves are presented in Fig. 5 for one uncoated and two coated Al-10 vol% SiC_w composites after an exposure at 750°C for 1 h. The Al-Si eutectic peak does not appear for the composite with coated SiC_w (40 nm). The Si content in the Al matrix as a function of exposure time at 750°C for uncoated and coated (40 nm) Al-10 vol% SiC_w is given in Fig. 6. The presence of the coating changes the reaction kinetics, e.g. there may exist an incubation time for the Al attack on coated SiC_w . This may be related to a low diffusivity of Al and of Si through complex layers of alumina at the beginning of the reaction process. After 15 min exposure the extent of reaction of Al with the coated SiC_w (40 nm) is only 1/5 of that with uncoated SiC_w . However, the protection effect of the coating is reduced with increasing exposure time length.

The protection effect of alumina coating (~15 nm) is also evident for SiC_p with a size of 17 μm . Fig. 7 shows DTA curves for Al-10 vol% SiC_p with uncoated and coated SiC_p after an exposure at 730°C for 15 min. The Al-Si eutectic peak is not visible for the composite with coated SiC_p . XRD data show that at this stage the extent of reaction in the composite with the coated SiC_p (15 nm) is about 1/3 of that with uncoated SiC_p . Further tests on Al- SiC_p are in progress.

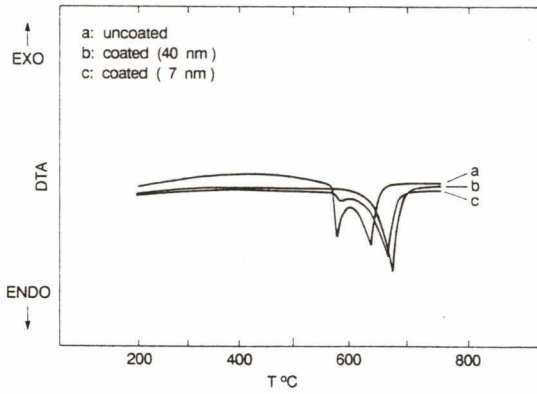


Fig. 5 DTA curves for Al-10 vol%SiC_w containing uncoated and coated (7 and 40nm) SiC_w after an exposure for 1 hour at 750°C.

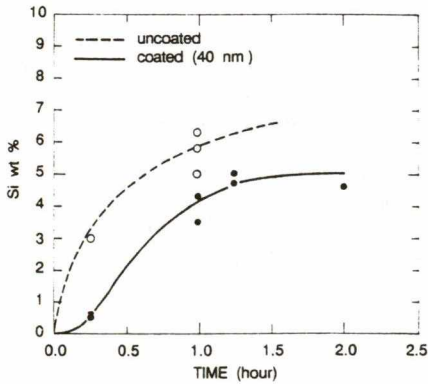


Fig. 6 Si content in the Al matrix as a function of exposure time at 750°C for uncoated and coated (40nm) Al-10 vol%SiC_w.

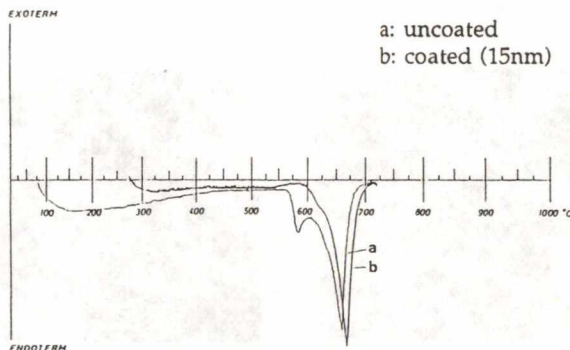


Fig. 7 DTA curves for Al-10 vol%SiC_p (17 μ m) containing uncoated and coated (15nm) SiC_p after an exposure for 15 min at 730°C.

5. THERMOMECHANICAL PROCESSING

In comparison to the conventional Al alloys, the Al-SiC composite materials have a more complicated response to the thermal and mechanical loadings as a result of the presence of large volume concentration of ceramic particles as well as the large differences in thermal and mechanical properties between the SiC reinforcement and the Al matrix. Fundamental studies of the microstructural and textural development during thermomechanical processing are of importance. To facilitate the microstructural observations, the present study is based on composites with low concentration of SiC. However, the effect of volume concentration will be discussed.

Cold deformation

The presence of SiC leads to an enhanced dislocation density in the annealed material prior to deformation. These are thermal dislocations generated during cooling from the processing temperature due to a large difference in thermal expansion coefficients between the SiC and the Al matrix [6]. During cold working the effect of SiC is to increase the dislocation density due to dislocation formation at the SiC [7]. As a result, the dislocation density is significantly greater and the cell size smaller in deformed Al-SiC composites compared to the SiC-free materials at a given strain. The enhanced rate of substructural development in Al-SiC composites during cold deformation may result in an increase in the amount of recovery, as observed in 90% cold rolled Al-2 vol% SiC_w in which subgrain walls are sharper than those in SiC_w-free material (Fig. 8).

The presence of SiC also gives a local effect on the deformation microstructure, e.g. many of the dislocations generated at the interface between the non-deformable SiC particle and the deforming matrix are localized and give rise to the formation of deformation zone of high dislocation density [8,9]. In deformation zones the subgrain size is smaller than that in the

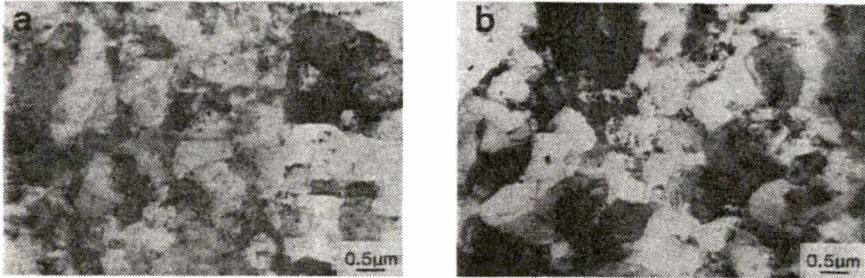





Fig. 8 TEM micrographs showing subgrains in 90% cold rolled (a) Al, and (b) Al-2 vol% SiC_w.

Table 5. Deformation zone in 90% cold rolled Al-SiC

	Zone shape	Distortion in metal flow	Zone size	Subgrain size	Misorientation
SiC _w		-	t = d	large	large
SiC _p (0.8 μm)		-	t = 0.5 d	large	small
SiC _p (3 μm)		+	t = 0.3-0.5 d	small	large

matrix and relatively large misorientations are often present. The microstructures and misorientations of deformation zones are related to the size and shape of SiC particles as well as to the strain. For equiaxed SiC particle, the larger the size the more strongly it influences the metal flow and the larger the misorientation in the deformation zone. Microstructural observations of deformation zones related to SiC_w, SiC_p (0.8 μm) and SiC_p (3 μm) are summarized in Table 5. SiC whiskers are very fine but have a large aspect ratio (~5) and sharp corners at the ends. Although they do not disturb metal flow strongly as SiC_p (3 μm) do, large misorientations are frequently found in the deformation zones at SiC_w ends and at SiC_w groups. The presence of SiC will cause a weakening of the deformation texture due to the lattice rotation within deformation zones [10]. This weakening effect depends on the shape, size and volume concentration of SiC and on the strain. Fig. 9 gives the cold rolling texture of Al materials without SiC_w and with 2 vol% SiC_w. The intensity of the rolling components decreases when the SiC_w are present. The texture weakening effect is likely to be important in composites containing high volume concentration of SiC [11].

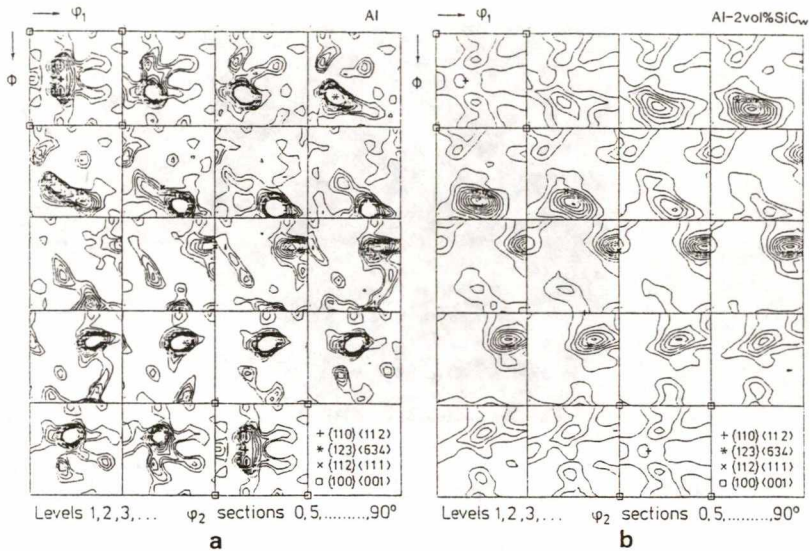


Fig. 9 90% cold rolling texture of (a) Al, and (b) Al-2 vol% SiC_w.

Heat treatment

During heat treatment of cold worked Al-SiC composites, the presence of SiC affects the recrystallization nucleation sites through its influence on cold deformation structures. The microstructural study shows that SiC larger than the critical particle size may stimulate nucleation in the deformation zones around them [9]. The strength of the SiC as nucleation sites is enhanced when they are present in groups (Fig. 10). SiC also influences the growth of nuclei by pinning of high-angle boundaries. The presence of fine Al₂O₃ particles does not essentially alter the nucleation sites. Their effect is pinning of dislocations and subboundaries and increasing the critical SiC particle size for nucleation. In the composites containing lower concentration of SiC, SiC particle stimulated nucleation and growth by high-angle boundary migration is dominating. Compared to SiC-free Al material the introduction of SiC leads to a faster nucleation rate, a lower recrystallization temperature and a finer recrystallized grain size, whereas the Al₂O₃ particles have the opposite effects (Table 6). Table 6 also shows the effect of the shape and size of SiC on recrystallization behaviour. It indicates that as nucleation sites, the SiC_p (0.8 μm) are weaker than SiC_w (higher recrystallization temperature and larger recrystallized grain size), and the SiC_p (3 μm) are similar to (after 90% cold rolling) and weaker than (after 50% cold rolling) SiC_w. This is in agreement with the observed differences in the deformation zone microstructure for the three reinforcements (see Table 5).

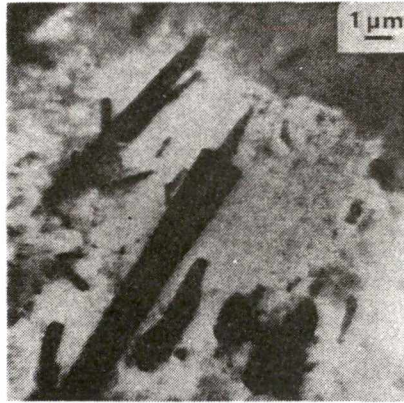


Fig.10 TEM micrograph showing a nucleus formed at a group of SiC_w in 90% cold rolled Al-2 vol% SiC_w heat treated at 294°C for 1 hour.

Table 6. Recrystallization temperature and grain size for cold rolled Al-SiC

	Cold rolling (%)	Recrystallization temperature (°C)	Recrystallization grain size* (μm)
Al-0.8% Al_2O_3	50 90	> 625 435	- 443
Al-0.8% Al_2O_3 - 2% SiC_w	50 90	470 325	622 154
Al-0.6% Al_2O_3 - 2% SiC_w	50 90	325 225*	377 { 140 3
Al-0.8% Al_2O_3 - 2% SiC_p (0.8 μm)	50 90	> 600 369	- 246
Al-0.8% Al_2O_3 - 2% SiC_p (3 μm)	50 90	> 600 329	- 198

* The grain size is the mean diameter of the equivalent circle grain in the rolling plane.

* The isochronal annealing curve does not have a definable recrystallization step.

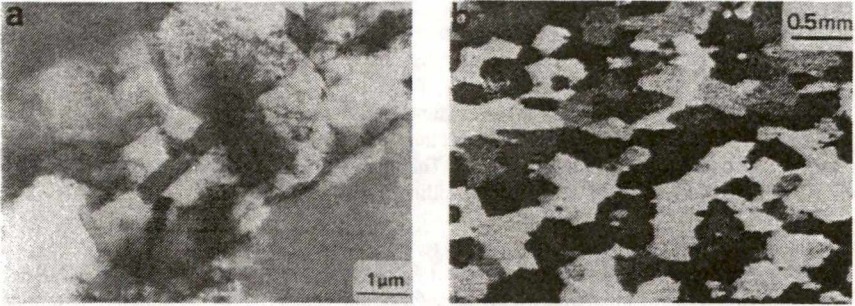


Fig. 11 (a) TEM micrograph of recovered microstructure of 90% cold rolled Al-10 vol%SiC_w heat treated at 600°C for 20 hours.
 (b) Optical micrograph of recrystallized microstructure of 90% cold rolled Al-2vol%SiC_w heat treated at 350°C for 1 hour.

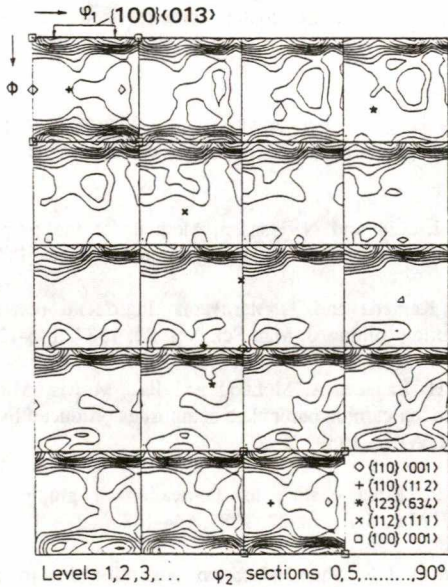


Fig. 12 Recrystallization texture of 90% cold rolled Al-2 vol%SiC_w.

With increasing concentration of SiC, the SiC are closely spaced. The particle spacing becomes smaller than necessary for the formation of nucleus. Instead of recrystallizing the composites recover on annealing. This process involves continuous growth of subgrains with increasing misorientations, and results in subgrain/grain of micron size. Typical recovered microstructure of 90% cold rolled Al-10 vol% SiC_w is shown by Fig. 11(a), which is very different from the recrystallized microstructure of 90% cold rolled Al-2 vol% SiC_w (Fig. 11(b)).

In Al-2 vol% SiC_w composite where SiC_w stimulated nucleation and growth by high-angle boundary migration is dominating during heat treatment, the recrystallization texture contains a strong component {100} <013> (Fig.12). This component is related to the rather strong shear component {100} <011> in the cold rolling texture through a ~20° rotation [12].

Microstructural study shows that in 90% cold rolled Al-2 vol% SiC_w, about 15% of the nuclei formed at deformation zones in the vicinity of SiC_w have orientation close to {100} <013>. It has been suggested that material within the deformation zones rotates to contain orientations close to {100} <013> and nuclei of this orientation may develop. In the fully recrystallized material, small grains are found to be of spread rolling, random or {100} <013> orientations, while most of the large grains have {100} <013> orientation, explaining the formation of the strong component {100} <013> in the recrystallization texture. In the composites containing high volume concentrations of SiC where the annealing involves continuous growth of subgrains, no reorientation through large angles can take place, the annealed texture is therefore expected to be weak and to contain some retained rolling components.

REFERENCES

1. B. Kindl, Y.L. Liu, E. Nyberg and N. Hansen, Control of interface and microstructure of the SiC/Al composites by sol gel techniques, Composites Science and Technology, in press.
2. B. Kindl, Y.L. Liu and N. Hansen, Method for the preparation of metal matrix composite materials, International Patent Application No. PCT/DK90/00172.
3. T. Iseki, T. Kameda and T. Maruyama, Interfacial reactions between SiC and aluminium during joining, J. Mat. Sci., vol. 22, 1987, 115-122.
4. D.J. Lloyd, H. Lagace, A. McLeod and P.L. Morris, Microstructural aspects of aluminium-silicon carbide particulate composites produced by a casting method, Mat. Sci. Eng., vol. A107, 1989, 73-80.
5. H.A. Katzman, Fibre coating for fabrication of graphite-reinforced magnesium composites, J. Mat. Sci., vol. 22, 1987, 144-148.
6. R.J. Arsenault and N. Shi, Dislocation generation due to differences between the coefficients of thermal expansion, Mat. Sci. Eng., vol. 81, 1986, 175-187.

7. M.F. Ashby, The deformation of plastically non-homogeneous materials, *Phil. Mag.*, vol. 21, 1970, 399-424.
8. F.J. Humphreys, The deformation structure and the recrystallization behaviour of two-phase alloys, *Metallurgical Forum*, vol. 12, 1978, 123-135.
9. Y.L. Liu, N. Hansen and D. Juul Jensen, Recrystallization microstructure in cold-rolled aluminium composites reinforced by silicon carbide whiskers, *Metall. Trans.*, vol. 20A, 1989, 1743-1753.
10. N. Hansen, D. Juul Jensen and Y.L. Liu, Effect of whiskers and small particles on the deformation and recrystallization texture of aluminium, *Proc. 9th Int. Conf. on Textures of Materials*, 1990, in press.
11. P.N. Kalu and F.J. Humphreys, Some aspects of the plasticity and texture of two-phase polycrystals, *Proc. 10th Int. Risø Symp., Risø National Laboratory, Roskilde, Denmark*, 1989, 409-414.
12. D. Juul Jensen, N. Hansen and Y.L. Liu, Texture development during recrystallization of an Al-SiC composite, *Proc. 10th Int. Risø Symp., Risø National Laboratory, Roskilde, Denmark*, 1989, 409-414.

