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EXPRESS REGULAR ARTICLE

Effect of Equal Channel Angular Pressing on the Distribution of Reinforcements in the Discontinuous Metal Matrix Composites

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The 6061 Al–10 vol% SiC_w composites were prepared by powder metallurgy with the powders having the different sizes, *i.e.* $< 30\,\mu m$ and $30\,\mu m <$. The composites were subjected to equal channel angular pressing (ECAP) under various conditions and the microstuctural changes during ECAP were examined. A special focus was made on the effect of ECAP conditions on the distribution of SiC whiskers. The present investigation was aimed at exploring the feasibility of ECAP as a post working process for manufacturing the discontinuous metal matrix composites. The microstructural examination and the microhardness measurement of the ECAPed samples suggested that the optimum combination of the uniform microstructure and enhanced mechanical properties would be obtained by (a) using the powders having the smaller size, (b) decreasing ECAP temperature, and (c) repeating ECAP.

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1. Introduction

Discontinuous metal matrix composites (MMCs), in which rigid discrete ceramic reinforcements are embedded in a ductile metal or alloy matrix, offer many advantages in applications due to their high specific mechanical properties compared to the unreinforced matrices. Of several routes processing the discontinuous MMCs including ingot metallurgy, high pressure or vacuum infiltration method, squeeze casting, *etc*,[1–5] powder metallurgy (PM)[6–8] is the most preferred one by: (a) ability producing the near net-shaped components, (b) easy control of the reinforcement volume fraction, and (c) suppression of chemical reaction at the interface between matrix and reinforcement due to relatively low processing temperature. In general, conventional metal working processes such as extrusion are performed on the as-PM processed discontinuous MMCs in order to obtain not only fully dense materials but the desired microstructures and mechanical properties. Since the mechanical properties of discontinuous MMCs are primarily dominated by that of the matrix and the characteristics of the reinforcement, *i.e.* volume fraction, shape, distribution, *etc,* it is essential to understand the microstructural evolution during the subsequent working processes following the PM process for effective fabrication of discontinuous MMCs.

Meanwhile, several advanced metal working techniques fabricating fully dense, ultrafine grained metallic materials with extraordinary high strength by imposing severe plastic deformation were recently developed.[9] They include equal channel angular pressing (ECAP),[10,11] severe torsional straining,[9] accumulative roll bonding,[12] *etc*. In spite of the successful application of severe plastic deformation to fabrication of unreinforced metals and alloys having a ultrafine grain structure and ultrahigh strength, no information is, at present, available on its application as a post working process of PM processed discontinuous MMCs. The present investigation was carried out to examine the feasibility of severe plastic deformation as a post working process of PM processed discontinuous MMCs. For this purpose, the ECAP technique was employed on the PM processed 6061 Al composite with 10 vol% silicon carbide whisker and the microstructural evolution during ECAP was examined. A particular focus was made on the effect of ECAP on the distribution and shape of SiC whisker. ECAP is, at present, the most developed severe plastic deformation technique producing bulk, porosity-free ultrafine grained materials. In ECAP, an ultrafine grained structure is obtained via mechanical fragmentation associated with severe plastic deformation by subjecting the sample to repetitive pressing into a die with the two equal cross-sectional channels intersecting at a certain angle.[10,11]

2. Experimental Procedures

The powders of 6061 Al having the two different sizes, one larger than $30\,\mu m$ and the other smaller than $30\,\mu m$, were prepared by the air-atomization method. The present 6061 Al had chemical composition of 1.01 Mg, 1.07 Si, 0.35 Cu, 0.25 Fe, 0.05 Mn, 0.12 Cr, and the balance Al in mass%. The 6061 Al powders were stir-mixed with SiC whiskers (SiC_w) having the average diameter and length of $\sim 0.45\,\mu m$ and $\sim 5\,\mu m$, respectively, under the stirring speed of 3000 rpm for 20 min such that SiC_w occupied 10 vol%. After hot pressing the mixture at 100 MPa and 773 K, the samples of $\phi 10\,mm \times 80\,mm$ were machined for ECAP. ECAP was conducted at temperatures of 373–673 K with a die designed to yield an effective strain of ~ 1 by a single pass: inner contact angle and the arc of curvature at the outer point of contact between channels of the die were 90° and 20°, respectively.

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In order to minimize the friction from the die wall, a MoS_2 lubricant was used. The detailed ECAP procedure was described elsewhere.[13,14] ECAP was performed up to 4 passes, *i.e.* accumulated effective strain of ~ 4, without rotating the sample between passages, *i.e.* route A.[15,16] In addition, some of the as-hot pressed composites made of the larger size powders were hot extruded at 673 K with an extrusion ratio of 12:1 before ECAP. The microstructures were examined by optical microscope (OM), field emission scanning electron microscope (FE-SEM, Jeol 6330F) and transmission electron microscope (TEM, Jeol 2010). Microhardness of the samples was measured using a Vickers microhardness tester at a load of 50 g for 15 s.

3. Results and Discussion

3.1 Distribution of SiC_w

3.1.1 Before ECAP

The optical microstructure of the composites before ECAP revealed the presence of the two distinct regions as shown in Fig. 1: the dark region of the SiC_w clusters and the white region of the alloy matrix. The clustering of short fibers during the P/M process of discontinuous MMCs was often reported previously.[17–19] An inspection of Fig. 1 revealed several findings. First, the shape of the SiC_w clusters was irregular in the as-hot pressed state. Second, their size and distribution were smaller and more uniform in the composite with the smaller initial powder size (Fig. 1(b)) compared to that with the larger initial powder size (Fig. 1(a)). Finally, as seen from the comparison between Figs. 1(a) and 1(c), hot extrusion resulted in the formation of SiC_w cluster stringers along the extrusion direction, more uniform distribution of the clusters, and fragmentation of the large clusters into several small ones. Figure 2 shows the FE-SEM micrographs of the SiC_w cluster region of the composites. In light of the fact that SiC was initially in the form of whisker with the average aspect ratio of ~ 10, it is of interest to note that a large portion of SiC in the clusters after hot pressing (Figs. 2(a) and 2(b)) or hot extrusion after hot pressing (Fig. 2(c)) was in the form of the particulate. As marked by arrows, this resulted from the breakage of the whiskers during hot working processes. In addition, SiC_w was densely packed and aligned along the extrusion direction in the clusters by hot extrusion.

3.1.2 After ECAP

(1) Alloy matrix

TEM microstructure of the alloy matrix after 2-passes ECAP at 373 K is presented in Fig. 3. The microstructure mainly consisted of parallel bands of elongated grains having the width of $\sim 0.3\,\mu$m and the length of $\sim 0.8\,\mu$m. The extended parallel band boundaries are often called the lamellar boundaries. The corresponding SAD pattern showed the diffused spots, indicating that the lamellar boundaries were mainly low angled. Inside the band interior, the dislocation cell boundaries (DBs) consisting of dense dislocation debris (marked by arrows) were also found. The dislocation density inside a cell enclosed by DBs was relatively low. DBs were typically normal to the lamellar boundaries. This kind of boundary structure was reported to be typical in the metals after heavy deformation.[19] It is worth mentioning that the present elongated grain structure resulted from the shear characteristics of the ECAP pattern used in this study, *i.e.* route A. For the route A in which no rotation was given to the sample between the pressing, the repetitive shear deformation is imposed on the same plane along the same direction. By contrast, when other routes, *i.e.* B_A, B_C and C, are used, the equiaxed grains are obtained by the 2 -passes or even number passes ECAP. In the routes B_A, B_C and C, the sample is rotated around its longitudinal axis between the passages by $\pm 90°$, $90°$ in the same direction and $180°$, respectively.[15,16] The grain refinement of the alloy matrix in the present composites is comparable to that reported on the unreinforced 6061 Al under the similar ECAP conditions.[20] This fact provides two important implications on manufacturing MMCs with ultrafine grained matrix: (a) the presence of discontinuous reinforcements does not affect the grain refinement of the soft matrix by intense plastic straining, and (b) the plastic deformation of MMCs is dominated by that of the matrix.

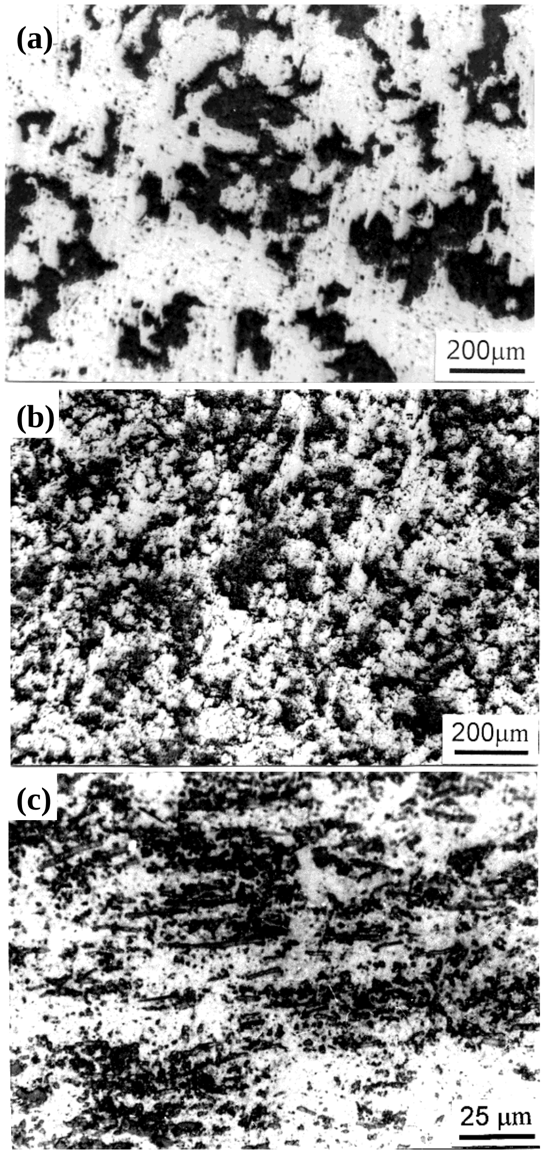

Fig. 1 The initial optical microstructures of the 6061 Al–10 vol% SiC_w composites before ECAP. (a) the larger initial powder size (30 μm <) (b) the smaller initial powder size (< 30 μm) (c) the as-hot extruded composite with the larger initial powder size (30 μm <).

(2) SiC_w clusters

The changes of the shape and distribution of SiC_w clusters

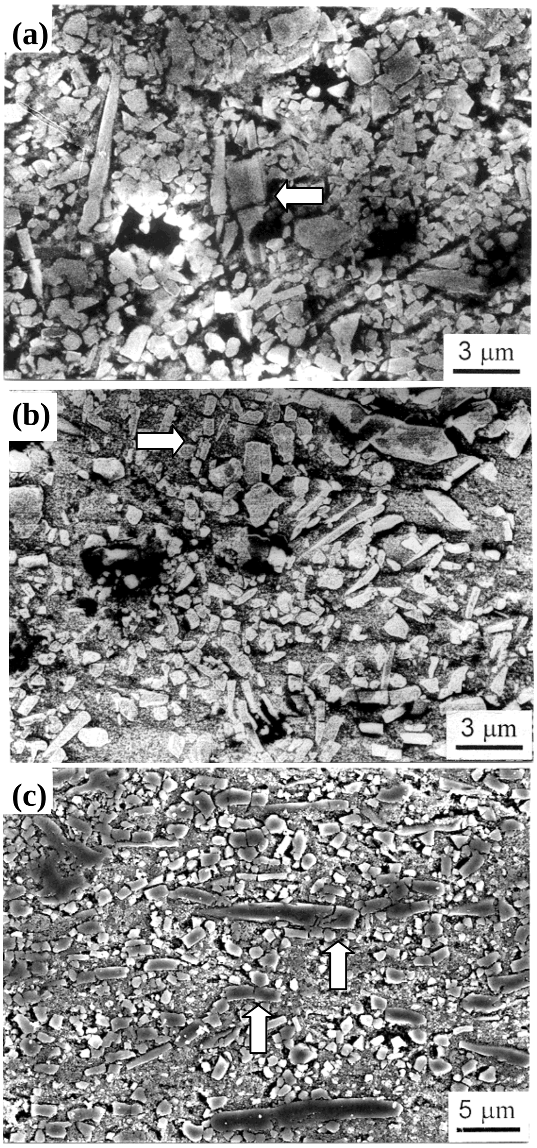

Fig. 2 FE-SEM micrographs showing the size and distribution of SiC_w in the cluster region. (a) the larger initial powder size (30 μm <) (b) the smaller initial powder size (< 30 μm) (c) the as-hot extruded composite with the larger initial powder size (30 μm <).

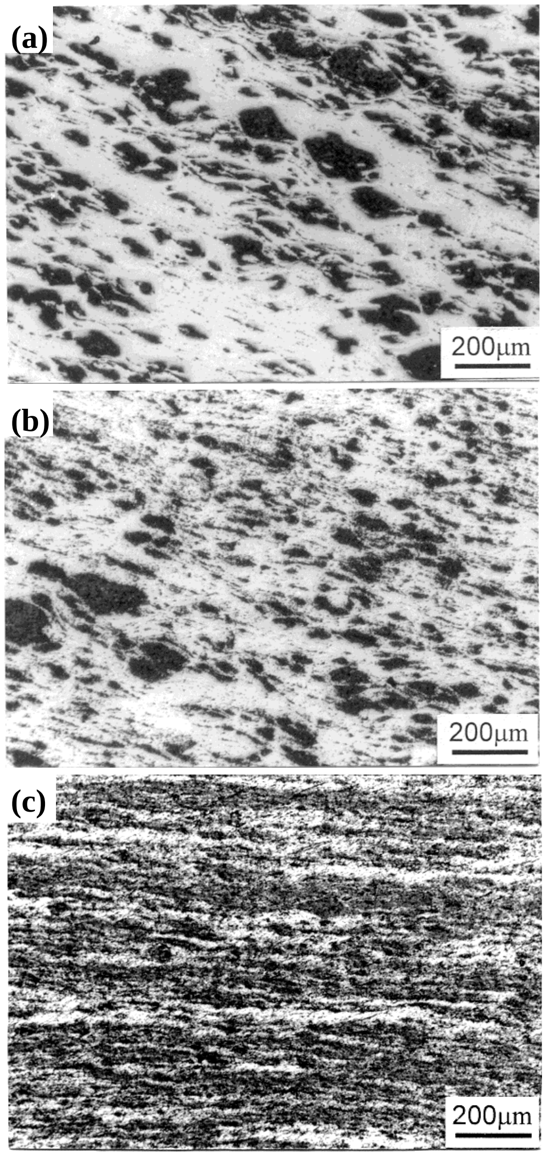

Fig. 4 Microstructural change of the 6061 Al–10 vol% SiC_w composite with the larger initial powder size (30 μm <) by ECAP at 373 K. The sample axis is horizontal. (a) a single pass (b) 2-passes (c) 4-passes.

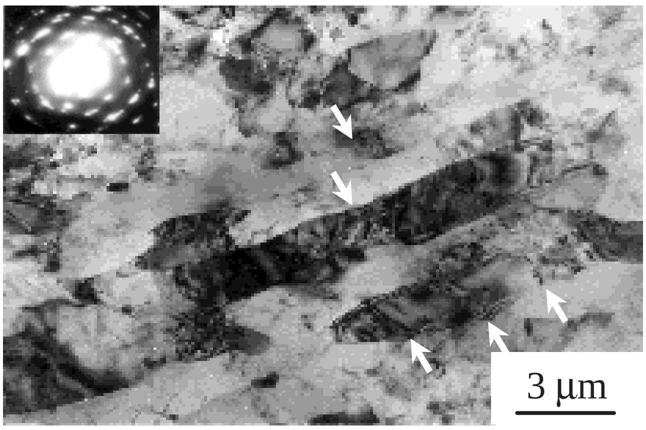

Fig. 3 TEM micrograph of 6061 Al matrix developed by 2-passes ECAP at 373 K.

in the composite with the larger initial powder size by repetition of ECAP at 373 K are shown in Fig. 4. By the first pass, the shape of the clusters changed from the initial irregular shape (Fig. 1(a)) to the ellipsoids with relatively sharp apices (Fig. 4(a)). The size distribution of the clusters was quite bimodal. The small ellipsoidal clusters with the length less than 50 μm and the large ones as long as 100 μm coexisted. The bimodal size distribution of the clusters would arise from the preferred separation of the branches of the initial clusters with irregular and jagged shape. In addition, the ellipsoidal clusters were aligned about 30° inclined to the longitudinal axis of the sample. This alignment is very close to the theoretical shear direction resulting from the present ECAP die, *i.e.* $\sim 26°$.[21] After the second pass (Fig. 4(b)), there was little change in the cluster shape but their size was reduced significantly. After 4-passes (Fig. 4(c)), the microstructure of the composite was manifested by unidirectionally well-distributed fine SiC_w clusters. Figure 5 shows the microstucture of the composite with the larger initial powder size de-

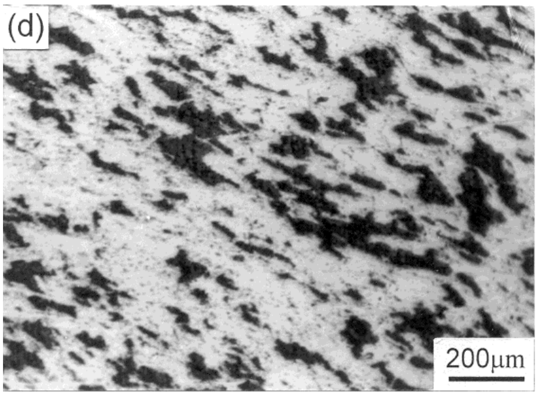

Fig. 5 Optical micrograph of the 6061 Al–10 vol% SiC_w composite with the larger initial powder size (30 μm <) after a single pass ECAP at 573 K.

veloped by a single pass of ECAP at 573 K. The comparison of Figs. 4(a) and 5 revealed that higher ECAP temperature resulted in more elongation of the SiC_w clusters due to enhanced plasticity of the matrix. The above description of the changes of the shape and distribution of SiC_w clusters in the composite with the larger initial powder size during ECAP was common in the composite with the smaller one. Accordingly, after 4-passes ECAP, the shape and distribution of SiC_w clusters in the latter were more fine and uniform, respectively, due to the smaller initial cluster size.

The microstructures of the composite with the larger initial powder size after 4-passes ECAP at 573 K following hot extrusion were shown in Fig. 6. Optically, ECAP after hot extrusion seemed to have little effect on the distribution of the SiC_w clusters (Figs. 1(c) and 6(a)). However, the comparison of the FE-SEM micrographs (Figs. 2(c) and 6(b)) revealed less dense packing of SiC whiskers in the clusters by conducting ECAP after hot extrusion.

3.2 Microhardness

The variation of microhardness of the composite with the larger initial powder size processed by a single pass of ECAP (without hot extrusion) is plotted as a function of ECAP temperature in Fig. 7. As seen in Fig. 4(a), a single pass resulted in heterogeneous microstructure and so the separate measurement was carried out on the alloy matrix and SiC_w clusters. By ECAP at 373 K, the microhardness of the matrix increased by ~ 50% due to the combined effects of strain hardening and grain refinement. However, only ~ 10% increase of the microhardness was obtained for the cluster region. It implies that no deformation occurred on SiC_w by ECAP and that shear deformation was not exerted effectively on the matrix in the cluster region during ECAP. This explanation is in harmony with the result of the microhardness measurement on the sample ECAPed at 473 K. By 473 K ECAP, the microhardness of matrix dropped significantly probably due to dynamic recovery while that of the clusters remained unchanged due to less or no effective plastic deformation on the matrix and SiC_w in that region. 573 K ECAP resulted in the microhardness drop at both regions. With increasing ECAP temperature, less dense packing of SiC_w in the clusters by the enhanced plasticity of the matrix and more progressive dynamic recovery of the matrix are expected. On the contrary to

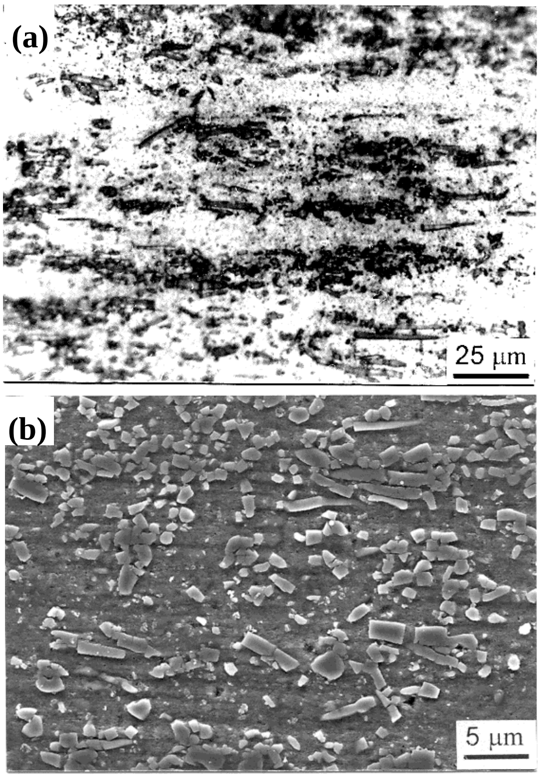

Fig. 6 The microstructure of the 6061 Al–10 vol% SiC_w composite with the larger initial powder size (30 μm <) after hot extrusion followed by 4-passes ECAP at 573 K. (a) optical (b) FE-SEM (SiC_w cluster region).

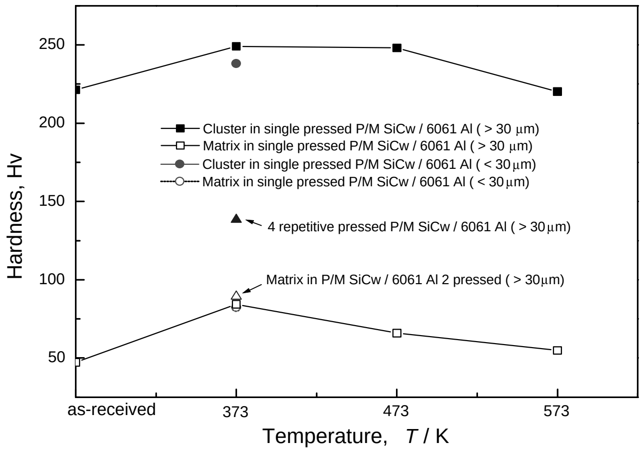

Fig. 7 The variation of the microhardness with ECAP temperature in a single pass ECAPed 6061 Al–10 vol% SiC_w composite with the larger initial powder size (30 μm <).

the single pass ECAPed sample, the microhardness of the 4-passes ECAPed sample was relatively uniform, $H_v = \sim 140$ at 373 K, throughout the sample by uniform distribution of SiC_w clusters (Fig. 4(c)). This value is very close to that reported on the 6061 Al–10 vol% Al_2O_3 composite produced by ingot metallurgy and 5-passes ECAP.[22,23] The results of the microhardness measurement described above for the composite with the larger initial powder size was consistently observed in the composite with the smaller initial powder size.

4. Conclusions

(1) The 6061 Al–10 vol% SiC_w composites were prepared by powder metallurgy with the powders having the different sizes. The composites were subjected to equal channel angular pressing (ECAP) under various conditions and the microstuctural changes were examined during ECAP in order to explore the feasibility of ECAP as a post working process for manufacturing the discontinuous metal matrix composites.

(2) The initial non-uniform microstructure of the composites consisting of the matrix and SiC_w clusters became more homogeneous with increasing the passages and temperature in ECAP. In addition, the smaller the initial powder size is, the more uniform distribution of the reinforcement was obtained.

(3) The optimum combination of the relatively homogeneous microstructure and enhanced mechanical properties of the composites, represented by the microhardness in this study, can be obtained by the three conditions of (a) the smaller initial powder size, (b) low ECAP temperature, and (c) repetition of ECAP.

Acknowledgements

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