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분말야금공정으로 제조한 SiC_w/6061Al 복합재료의 미세조직에 미치는 ECAP가공의 영향

장 시 영
한국항공대학교 항공재료공학과

Effect of ECAP on Microstructure of SiC_w/6061Al Composites Produced by Powder Metallurgy

Si-Young Chang
Department of Materials Engineering, Hankuk Aviation University, Kyonggi-do 412-791, Korea
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Abstract The 6061 Al alloy based composites reinforced with 10 vol% SiC whiskers were prepared by powder metallurgy with the powders having the different sizes, i.e. <30 μm and >30 μm. The composites were subjected to equal channel angular pressing (ECAP) at various conditions and the microstructural changes during ECAP were examined. In the composites, SiC whiskers were clustered and randomly aligned. The clusters were relatively well distributed in the composite with the smaller initial powder size. After ECAP, the clusters were aligned parallel to flow direction and became smaller. In addition, the shape of clusters was changed from irregular to round. The microstructure of the ECAPed samples were compared with those of the conventionally hot-extruded composites. The uniform microstructure and enhanced microhardness could be obtained by using the powders having the smaller size, decreasing ECAP temperature and repeating ECAP.

Keywords : Equal Channel Angular Pressing, 6061Al, Powder Metallurgy, SiC whisker, Composite

1. Introduction

Metal matrix composites (MMCs) have received much attention as potential structural materials for their high specific strength and stiffness. Such MMCs are expected in high performance applications such as in aircraft and automotive parts. In order to obtain the higher specific mechanical properties for better performance, in particular, MMCs reinforced with short fibers have been developed and generally produced by squeeze casting[1-5] and powder metallurgy (P/M).[6-8] During the high pressure infiltration in the squeeze casting process, the reinforcements have a tendency to be readily damaged. And also, reaction products could be generated at the reinforcement/whisker interface due to the high infiltration temperature. As a result, it is very hard to obtain the sound composites with ideally increased strength. In contrast, the powder metallurgy has been used for producing composites because of the several merits that are possible to reduce the reaction at the interface due to the mixing at lower temperature than melting temperature, to produce the near net-shaped components and to control easily the volume fraction of reinforcements. Following the powder metallurgy process, in general, the subsequent processes such as rolling, extrusion and forging has been performed for further improvement of the mechanical properties of P/M MMCs through the grain refining of microstructure, unidirectional alignment of reinforcements and improvement of bonding of matrix/reinforcement interfaces.

Equal channel angular pressing (ECAP) technique has been used as an attractive method to obtain a sub-micrometer ultra-fine grained (UFG) structure, because it gives large bulk metallic materials without residual porosity.[9-13] Recently, Valiev et al.[14] and Li et al.[15] applied the ECAP technique for grain refining and improvement of mechanical properties of 6061 Al alloy matrix com-posite reinforced with Al_2O_3 particulates fabricated by casting technique. However, report on applying the ECAP technique as the subsequent process following the powder metallurgy of the MMCs is deficient. Accordingly, it is of interest to examine the effect of simple shear deformation during the ECAP on the alignment and distribution of reinforcements in MMCs.

In this work, therefore, in order to evaluate the feasibility of ECAP as the subsequent process following the powder metallurgy process for fabrication of MMCs, the 6061 Al alloy matrix composites reinforced with SiC whiskers were prepared by powder metallurgy, and the effect of ECAP on microstructure and microhardness of P/M SiCw/6061 Al composites was investigated.

2. Experimental procedure

Two kinds of air atomized 6061 Al alloy (Al-1.01%Mg-1.07%Si-0.35%Cu-0.25%Fe-0.05%Mn-0.12%Cr (in wt.%)) powders; one has a size above 30 μm and the other has a size below 30 μm, were prepared, and then were mixed with SiC whiskers with an average diameter of 0.45 μm and a length of ~5 μm by stirring under a stirring speed of 3000 rpm for 20 min. The obtained powders with 10% in the volume fraction of SiC whiskers were hot-pressed at 773 K under a pressure of 100 MPa. The hot-pressed samples with a dimension of $\Phi 32 \times 80$ mm were machined to cylindrical samples of $\Phi 10 \times 80$ mm as-received materials for ECAP. The ECAP was carried out using a press speed of 2 mms^{-1} with MoS_2 as a lubricant at 373~673 K. The present ECAP die was designed to yield an effective strain of 1 by a single press: the inner angle and the arc of curvature at the outer point of contact between channels of the die were 90° and 20°, respectively.[16] During ECAP, the samples were repetitively pressed using processing route A in which the sample is repetitively pressed without any rotation[17]. In addition, a part of as-received sample was hot-extruded at an extrusion ratio of 12:1 and at 673 K followed by ECAP.

Microhardness was measured using a Vickers microhardness tester at a load of 0.05 kg for 15 s. Microstructure examination of samples before/after ECAP was carried out using optical microscope (OM), field emission scanning electron microscope (FE-SEM, JSM6330F, JEOL, Japan) and transmission electron microscope (TEM, JEOL 2010, Japan) operated at 200 keV.

3. Results and Discussion

3.1 Microstructural characteristics

Figure 1 shows the typical microstructure of as-received P/M SiCw/6061 Al composites before ECAP. The as-received P/M SiCw/6061 Al composites revealed the microstructure consisting of two distinct regions; one is the black region of the clusters of SiC whiskers and the other is the white region representing 6061 Al matrix. The clustering

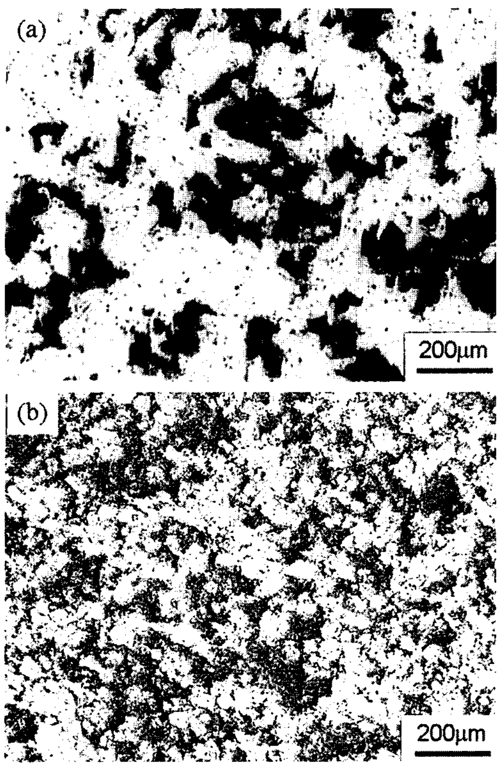

Fig. 1. Optical micrographs of the P/M SiCw/6061 Al composites before ECAP; (a) >30 μm and (b) <30 μm in Al powder size.

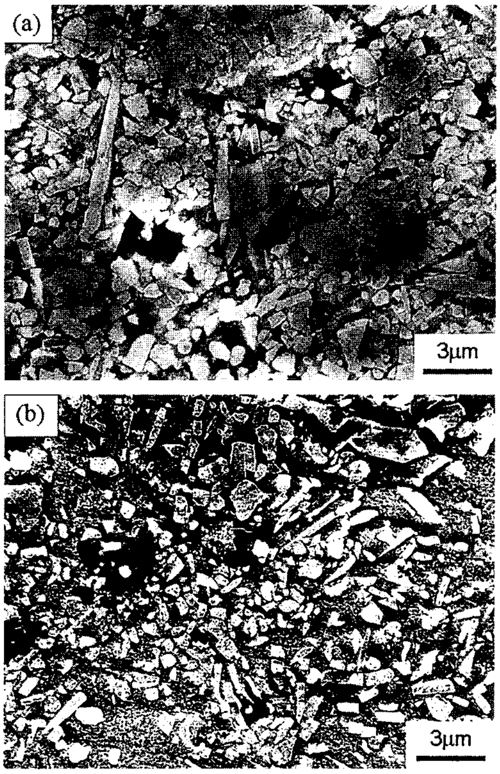

Fig. 2. FE-SEM micrographs of the cluster in the P/M SiCw/6061 Al composites before ECAP; (a) >30 μm and (b) <30 μm in Al powder size.

of short fibers during the P/M process of discontinuous MMCs was often reported previously.[18-20] The clusters were relatively well distributed in the composite with the smaller initial powder size. Figure 2 represents the highly magnified FE-SEM micrographs of the cluster in the composites. The composites with the smaller initial powder size revealed that the SiC whiskers were aggregated more densely, compared with that with the larger initial powder size. On the other hands, the SiC whiskers were randomly distributed in the cluster. 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 cluster after hot pressing (Fig. 2) or hot extrusion after hot pressing (Fig. 8a) was in the form of the particulate. This resulted from the breakage of the whiskers during hot working process.

The optical microstructures of ECAPed P/M SiCw/6061 Al composites are shown in Fig. 3. Unlike the extruded P/M SiCw/6061 Al composite (Fig. 7), the clusters still remained after ECAP. However, they were distributed more homogeneously and the shape changed from the large irregular island shape to the small round island shape after ECAP. These microstructural characteristics became stark with repeating ECAP and decreasing ECAP temperature. In addition, the clusters were aligned 30^o 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. $\sim 26^o$.[21] In the composite with the smaller initial powder size, the homogeneous microstructure could be obtained by ECAP as shown in Fig. 4. In particular, the 4 repetitively ECAPed composite at relatively low temperature of 373 K showed that the large clusters disappeared and the SiC whiskers were relatively well distributed. The alignment and distribution of SiC whiskers in the cluster were shown in Fig. 5. The SiC whiskers in the cluster that were densely aggregated before ECAP became slightly distant after ECAP. This tendency obviously appeared with repeating ECAP. However, the random distribution of SiC whiskers in the cluster remained almost unchanged.

TEM microstructure of matrix in the ECAPed composite was represented in Fig. 6. After 2 ECAP at 373 K, the grains having an initial size of approximately 20 μm were elongated and had a length of ~0.8 μm and a width of ~0.3 μm. It is apparent from the selected area diffraction pattern showing the diffused spots and the extra spots that the microstructure consists of many small grains having random distribution of orientation. 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. 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.[22] From these facts, it is also apparent that the grain size is very much reduced by ECAP, even if the size, alignment and distribution of SiC whiskers appear to be relatively unaffected as shown in Fig. 5, indicating that the plastic deformation of MMCs is dominated by that of the matrix.

The P/M SiCw/6061 Al composite was subjected

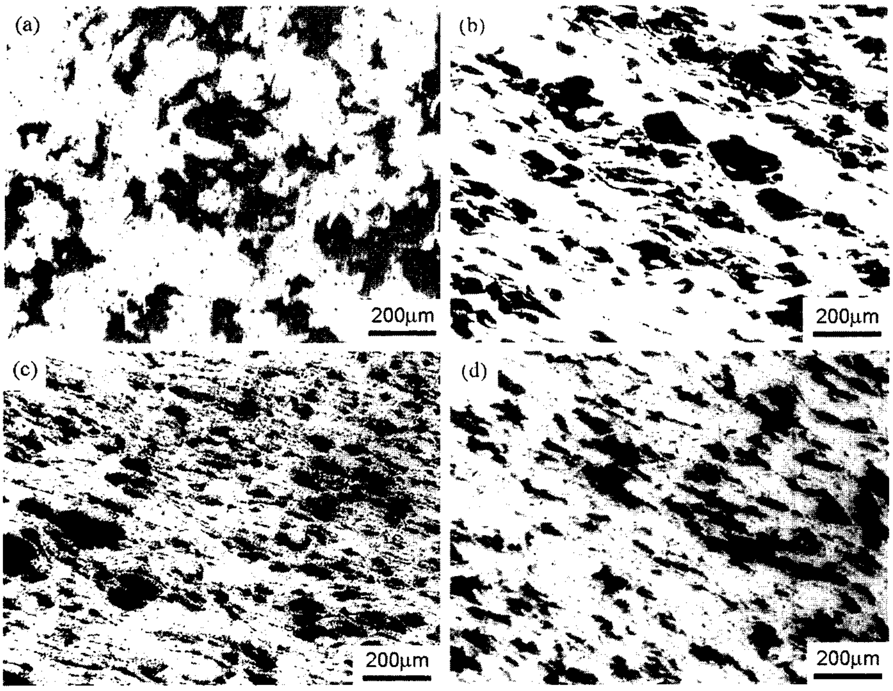

Fig. 3. Optical micrographs of the ECAPed P/M SiCw/6061 Al composites (>30 μm in Al powder size); (a) 0 ECAP, (b) 1 ECAP at 373 K and (c) 2 ECAP at 373 K, (d) 1 ECAP at 573 K.

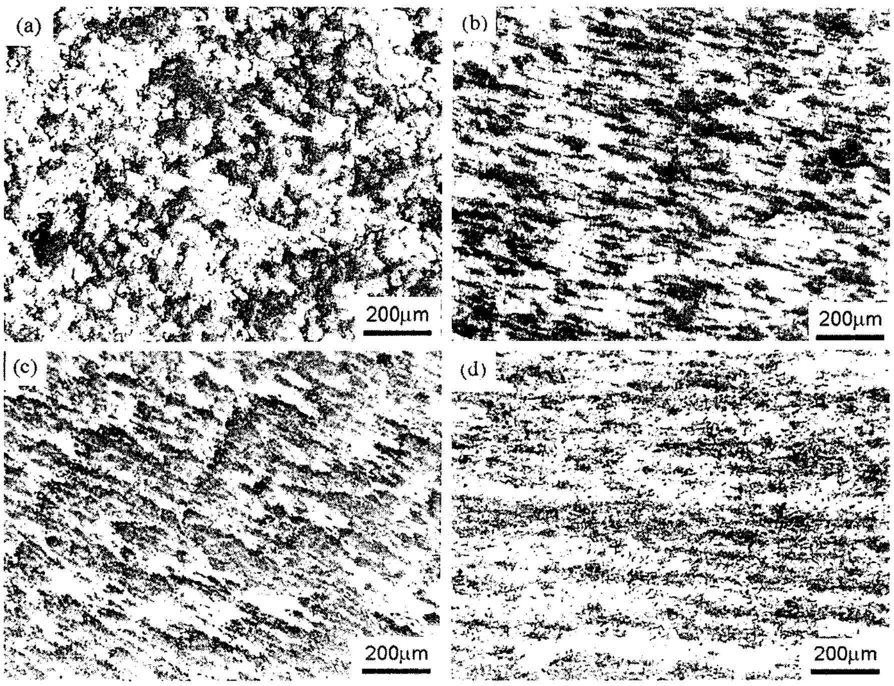

Fig. 4. Optical micrographs of the ECAPed P/M SiCw/6061 Al composites (<30 μm in Al powder size); (a) 0 ECAP, (b) 1 ECAP, (d) 4 ECAP at 373 K, (c) 1 ECAP at 473 K.

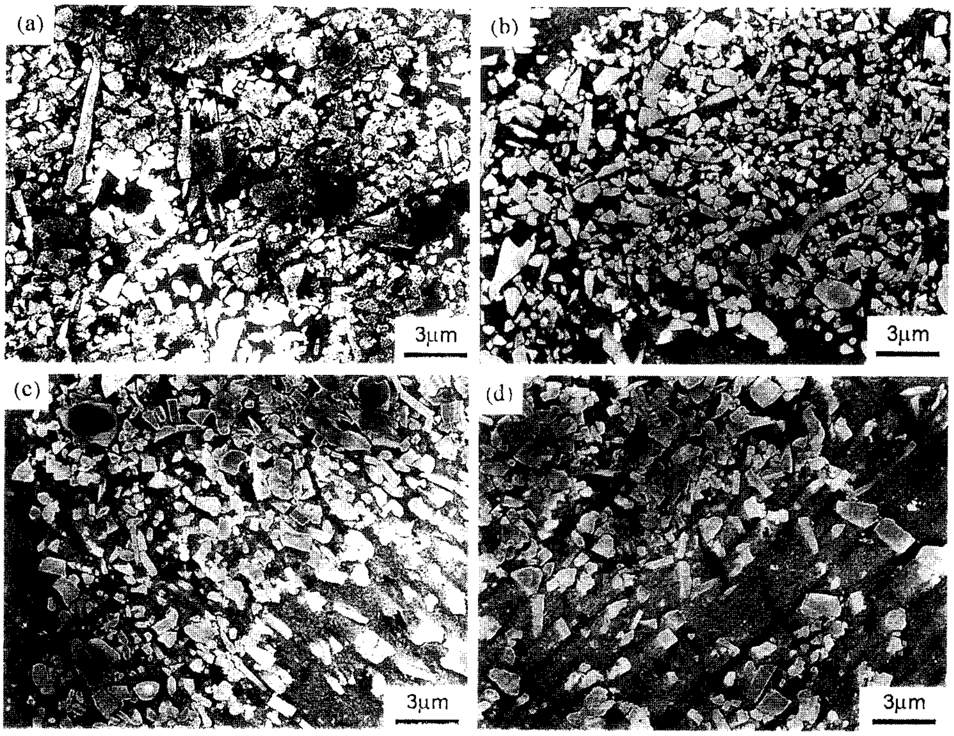

Fig. 5. FE-SEM micrographs of the cluster in the ECAPed P/M SiCw/6061 Al composites (>30 μm in Al powder size); (a) 0 ECAP, (b) 1 ECAP, (d) 2 ECAP at 373 K, (c) 1 ECAP at 473 K.

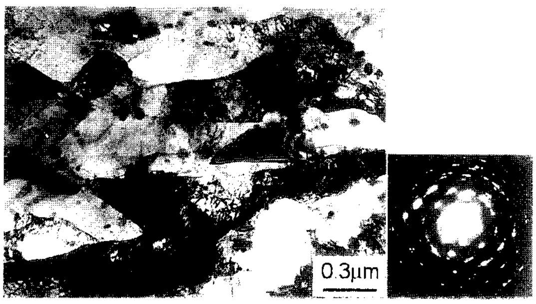

Fig. 6. TEM micrograph of matrix in the P/M SiCw/6061 Al composite (>30 μm in Al powder size) after 2 repetitive ECAP at 373 K.

to the conventional hot extrusion followed by ECAP, in order to investigate the ECAP effect on the microstructure of the extruded composite. Figure 7 reveals the microstructures of the extruded P/M SiCw/6061 Al composite before/after ECAP. The flow direction during ECAP is left to right, which is same with the extrusion direction. In the as-extruded composite, there was no cluster of the SiC whiskers. The SiC whiskers were relatively well distributed and aligned parallel to the extrusion direction. After ECAP, the array of SiC whiskers became improved slightly along to flow direction. This tendency was stark with increasing ECAP temperature and number of ECAP. This result could be confirmed by highly magnified observation using FE-SEM as shown in Fig. 8. Thus, the reason why the SiC whiskers aligned parallel to extrusion direction was well realigned along to flow direction during ECAP is considered to be due to that matrix which is weaker than SiC whiskers flows preferentially during ECAP, and the matrix flow occurs readily with increasing ECAP temperature and repeating ECAP.

3.2. Microhardness

The microhardness of the ECAPed P/M SiCw/6061 Al composites is shown in Fig. 9. The as-received P/M SiCw/6061 Al composite prepared with the powders having the larger size, >30 μm, had different microhardness between the cluster and

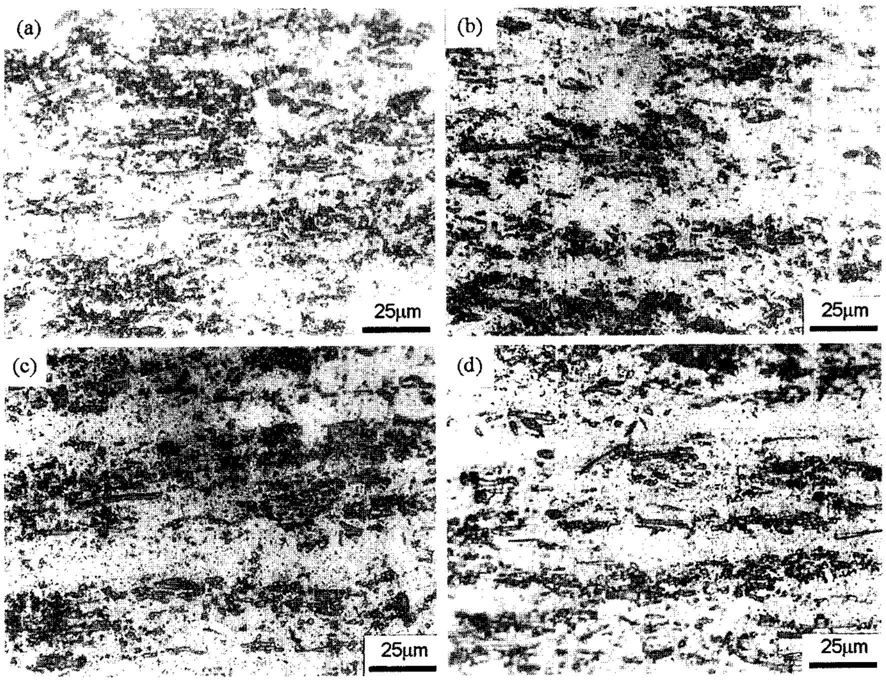

Fig. 7. Optical micrographs of the extruded P/M SiCw/6061 Al composites (>30 μm in Al powder size) after ECAP; (a) as-extruded, (b) 1 ECAPg at 473 K, (c) 1 ECAP at 573 K, (d) 4 ECAP at 673 K.

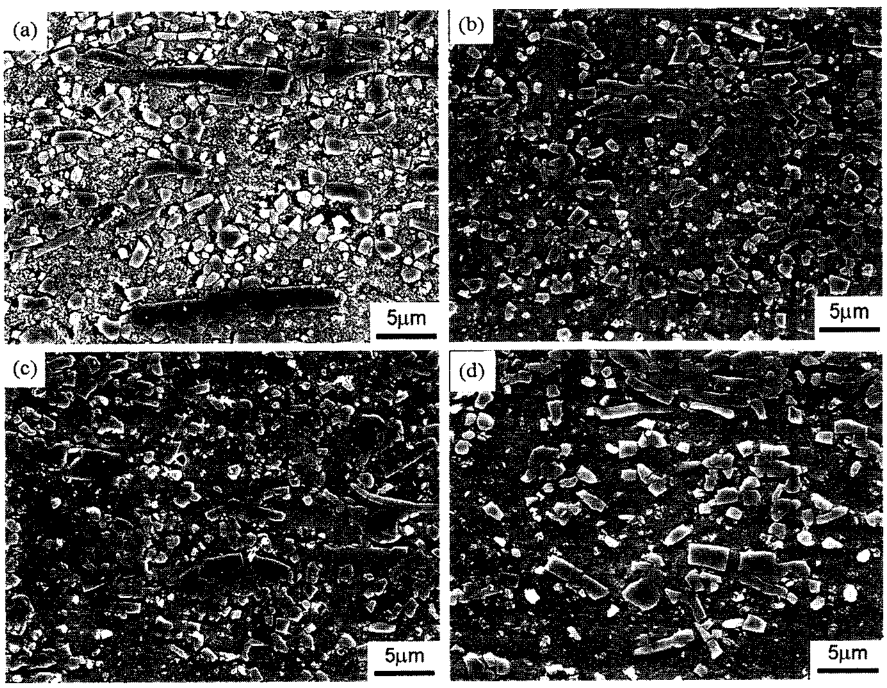

Fig. 8. FE-SEM micrographs of the extruded P/M SiCw/6061 Al composites (>30 μm in Al powder size) after ECAP; (a) as-extruded, (b) 1 ECAP at 473 K, (c) 1 ECAP at 573 K, (d) 4 ECAP at 673 K.

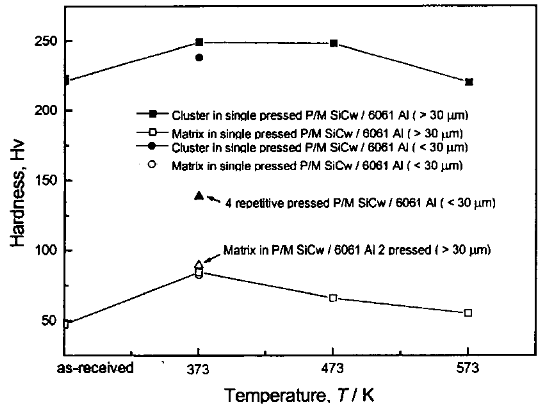

Fig. 9. Microhardness of the ECAPed P/M 6061 SiCw/ 6061 Al composites.

matrix. The microhardness in the cluster and matrix increased respectively after a single ECAP, and slightly decreased with increasing ECAP temperature. This was comparable to the recent reports that the yield stress in Al alloys[23)] and tensile strength in a low carbon steel[24)], which were produced by conventional casting followed by rolling, decreased with increasing ECAP temperature. In addition, the microhardness of matrix in the 2 repetitively ECAPed composite was higher than that of single ECAPed one, but its increment was small. In general, the increase of microhardness in the matrix after ECAP is because of the work hardening that is caused by the formation of subgrain bands and the density increase of dislocation occurring with the shear deformation in the initial grain interior. Additionally, the reason why the microhardness decreases with increasing ECAP temperature is deduced to be due to the dynamic recrystallization occurring during ECAP[25)] at higher temperatures. The composite prepared with the powders having the smaller size also showed the separated microhardness between matrix and cluster. However, the microhardness of the 4 repetitively ECAPed smaple was relatively uniform, resulting from the microstructure with the relatively well-distributed SiC whiskers as shown in Fig. 4d). This value is very close to that reported on the 6061 Al-10 vol.% Al_2O_3 composite produced by ingot metallurgy and 5 repetitive ECAP.[26,27)] It is apparent from the above results that the repetitive ECAP is sufficient to give the homogeneous microstructure and high

Table 1. Microhardness of the extruded P/M SiCw/6061 Al composites (>30 μm in Al powder size) after ECAP.

	Hardness (Hv)
As-received	134
1 ECAP at 473 K	139
1 ECAP at 573 K	146
2 ECAP at 673 K	131
4 ECAP at 673 K	154

microhardness in the P/M composites with the smaller initial powder size. Consequently, it is concluded that the ECAP technique can be used as the subsequent process following the powder metallurgy of discontinuous MMCs.

The microhardness of the extruded P/M SiCw/ 6061 Al composite followed by ECAP was listed in Table 1. The extruded composite showed uniform microhardness, unlike the as-received and ECAPed composites. The microhardness increased slightly after ECAP. In particular, the microhardness of composite after ECAP at 573 K was slightly higher than that after ECAP at 473 K. This is deduced to be due to the array of SiC whiskers that becomes a little better according to the flow direction as shown in Fig. 8. In contrast, the microshardness decreased at higher ECAP temperatures than 573 K. This is interpreted by the weakening due to dynamic recrystallization of matrix, which indicates that it overcomes the hardening effect due to the improved alignment of SiC whiskers caused by matrix flow during ECAP. However, even at such high ECAP temperatures, for example 673 K (Table 1), the repetitive ECAP could cause the increase of microhardness. This indicates that the microhardness would be increased by the repetitive ECAP causing further improvement of alignment of SiC whiskers despite the dynamic recrystallization at high ECAP temperatures. However, the increase of microhardness in the extruded composite after ECAP was totally small compared with the as-received one.

4. Conclusions

For the 6061 Al alloy based composites reinforced with 10 vol% SiC whiskers prepared by powder metallurgy, the microstructural changes during the ECAP were examined and the feasibility of ECAP as the subsequent process following powder metallurgy of the discontinuous MMCs was evaluated.

In the composites, SiC whiskers were clustered and randomly aligned. After, however, the clusters of SiC whiskers remained unchanged and were aligned parallel to the flow direction and well distributed. In addition, the shape of clusters was changed from irregular to round. This microstructural evolution became stark with repeating ECAP and decreasing ECAP temperature. The uniform microstructure and microhardness could be obtained in the composites with the smaller initial powder size after the repetitive ECAP at low temperature, which is comparable to those of the conventionally hot-extruded composite.

The ECAP technique can be effectively applied as the subsequent process after powder metallurgy of the discontinuous MMCs, in particular, the repetitive ECAP at relatively low temperatures are sufficient to give the homogeneous microstructure of well-distributed SiC whiskers resulting in high microhardness.

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