Fabrication of Two-Layered Al-B_4C Composites by Conventional Hot Pressing Uuder Nitrogen Atmosphere and Their Characterization

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In this study, we describe the conventional hot pressing (CHP) of layered Al-B_4C composites and their characterization. The matrix alloy Al-5 wt.%Cu was prepared from elemental powder mixtures. The metal and B_4C powders were mixed to produce either Al-Cu-10vol.%B_4C or Al-Cu-30vol.%B_4C combinations. Then, these powder mixtures were stacked as layers in the hot pressing die to form a two-layered composite. Hot pressing was carried out under nitrogen atmosphere to produce $30 \times 40 \times 5$ mm specimens. Microstructural features and age hardening characteristics of composites were determined by specimens cut longitudinally. The flexural strength of both layered composites and their monolithic counterparts were investigated via three point bending tests. In the case of layered specimens of both 10vol.%B_4C and 30vol.%B_4C containing layers were loaded for three-point test. The results show that a homogeneous distribution of B_4C particles in the matrix alloy which is free of pores, can be obtained by CHP method. The ageing behavior of the composites was found to be influenced by the reinforced materials, i.e. higher hardness values were reached in 8 hrs for the composites than that for the matrix alloy. Flexural strength test showed that two-layered composites exhibited improved damage tolerance depending on layer arrangement. Microstructural investigation of the fracture surfaces of the bending specimens was performed by means of scanning electron microscope (SEM). While layer with lower reinforcement content exhibited large plastic deformation under loading, the other with higher reinforcement content exhibited less plastic deformation.

Key Words : Ceramic Reinforced Al Metal Aatrix Composite, Hot-Pressing Method, Age Hardening, Flexural Strength, Fracture Surface

1. Introduction

In the last decade, as demand for high quality materials are increased, the development of lightweight aluminum (Al) alloys also increased especially in aerospace and automotive industries (Soma, 2003). It has been well known that Al-based metal matrix composites (MMCs) offer a very low thermal expansion coefficient, high specific strengths, wear and heat resistance as compared to conventional Al alloys (Lloyd, 1994; Nitsham, 1997; Ibrahim, 1991). In order to combine all these properties, MMCs have become a very attractive method for various industrial applications (Reihani, 2006).

Powder metallurgy (PM) and liquid state producing (LSP) techniques are known to produce MMCs. The advantage of PM is that it can produce Al-MMCs, in which particles are distributed homogeneously with a density close to the theoretical values (Monastyrsky, 2002; Yong, 2002; Kawamura, 2002; Ivasishin, 2002; Moustafa, 2002;

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Vinicius, 2001 ; Radhakrishna, 2002). PM technique is too complex and requires several steps, i.e. mixing and blending of pre-alloyed powder with reinforcement powder, degassing under vacuum, consolidation (hot pressing or HIP) and secondary processing (extrusion or rolling). However, any additional step in the production of Al-MMCs increases the cost and hampers its commercial success.

However, LSP technique is more advantageous due to its lower cost and higher performance (Kevorkijan, 1998 ; Ramesh, 1999). This technique offers more flexibility and can be divided into three groups such as compo-casting, rheo-compocasting, and squeeze casting (Howes, 1986 ; Harris, 1988 ; Alonso, 1993 ; Suresh, 2003). The compo-casting method is a casting method which requires the addition of ceramic particles in the molten metal, followed by stirring and casting. The rheo-compocasting method is a rapid solidification process, which consists of re-melting and instantaneous solidification. The squeeze casting method is also common manufacturing process for fabrication of composite. These techniques generally present similar problems as, (a) non-uniform distribution of ceramic particles due to the agglomeration and dendritic segregation, (b) undesirable chemical reaction at the interface due to the high temperature of the melt and (c) negative pressure occurring during the stirring to create a vortex (Kim, 1994 ; Kanetake, 1994 ; Sahin, 2003)

Here, we propose an alternative production method for particulate reinforced composites. Hot pressing of powders with a small amount of liquid phase is used as a simple and less complicated consolidation method to fabricate Al-MMCs with homogeneously dispersed particles without porous structures. By this method, advantages of both powder metallurgy and liquid state production techniques are benefited. Fully dense metal powder compacts with controlled microstructures can be produced by hot consolidation in which pressure and heat are applied simultaneous rather than sequentially, as in conventional P/M processing (Kaya, 1999). Our previous studies indicated that although the matrix alloy was prepared from elemental Al and Cu powders and the powder mixtures were conventionally hot pressed under nitrogen atmosphere, composites comprised of 0-30 vol.% ceramic reinforcing particulates, yielded reasonable mechanical properties (Bedir, 2001 ; 2002 ; 2005).

On the other hand, in order to further improve the mechanical properties of the particulate reinforced composite systems, the layered configuration is being adopted as an alternative metal/ceramic composite. The main advantages of such a material system are the in-plane isotropy of both strength and modulus, relatively high specific strength and modulus, high toughness and a relatively low cost due to a simpler processing route. However, all the above properties depend on the architecture of the composite (Sherman, 1998).

In the present study, CHP method was extended to producing of two-layered Al-B_4C composites in order to increase its mechanical strength and define the role of the residual stress in the interface.

2. Experimental Procedure

The chemical composition of matrix alloy used in this study is Al-5wt.%Cu, and consist of pure Al powder (purity 99.9%, <25 μm) and pure Cu powder (purity 99.9%, <63 μm). Cu powder is added to the mixture to provide formation of the liquid phase and densification during the hot pressing. Three different composite mixtures were prepared by mixing B_4C powder (purity 99%, <10 μm) of 10vol.%, 20 and 30 separately, over matrix alloy with addition of a small amount isotropic alcohol as shown in Table 1. The composite powder was either filled to form a single

Table 1 Chemical composition of the composites

Composite	Explanation of the composition (in volume)	
Al-10B_4C	90%Al-Cu	10%B_4C
Al-20B_4C	80%Al-Cu	20%B_4C
Al-30B_4C	70%Al-Cu	30%B_4C

Note : The matrix alloy of the composite Al-Cu was 95% Al and 5% Cu by weight.

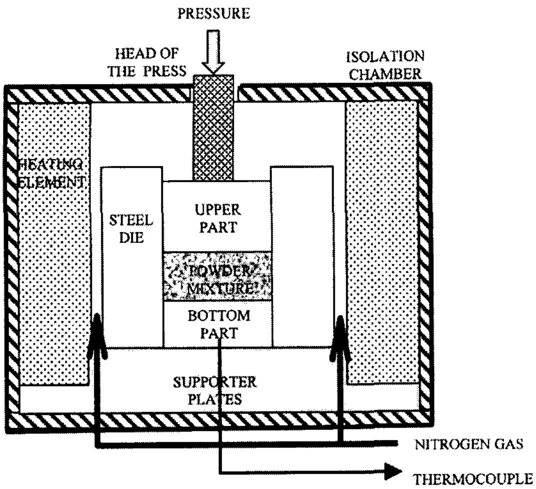

Fig. 1 Schematic cross-section of the hot press

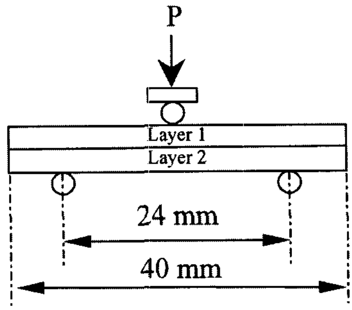

Fig. 2 The schematic illustration of specimen-replacement for bending test

layer or stacked as layers in to a uniaxial die, made of X40CrMoV51 (AISI H13) hot work tool steel having a rectangular cross-section of 30 by 40 mm, as shown in Fig. 1.

In the single layer case, powder mixture in the die is hot consolidated by pressure and heat applied simultaneously. In two-layered case, the first powder mixture is cold consolidated in the die but afterwards the second layer is placed on top and both layers are hot consolidated. Cold pressing was at 40 MPa pressure and hot pressing was at 600℃ (rising at a rate of 20℃/min.) with 25 MPa constant pressure under nitrogen atmosphere. In order to avoid porous structure in the composite the pressure on the specimen was not released along 5 minutes until consolidation was finished followed by cooling specimen down to 350℃.

To determine the age-hardening behavior of composites, the specimens which were cut from composite plate, were treated at 530℃ for 24 hrs. The specimens were transferred into an oil bath and age-hardened at 180℃ for various ageing periods of 2, 4, 6, 8, 10, 12, 16, 20 and 24 hours after cold water quenching. Hardness values of the specimens were subsequently determined with HB at a load of 62.5 kg and SEM was used to examine the distribution of the ceramic particles in the composite-microstructures. Metallographic preparation of the specimens was done with 600 and 1200 grit papers and polished with 3 μm diamond paste.

The flexural strength measurement was performed using a three-point bending test on the specimens with 10 mm thickness and 5 mm width where the loading span was 24 mm. The schematic illustration of bending test apparatus which was designed for this research, is shown in Fig. 2. Each surface in the length direction was ground with SiC paper and one prospective tensile surface for three-point bending tests was finally polished with diamond slurry having an average particle size of 3 μm. The flexural strength was calculated from the following formula;

$$\sigma_{b3}=\frac{3PL}{2wt^2}$$

where P is the maximum load causing fracture, L is the span, w and t are the width and thickness of the specimen, respectively. The fracture surfaces of the specimens were examined by SEM after bending test.

3. Results and Discussion

3.1 Microstructure observation

The aim of this study was to produce particle-reinforced composite without pores by using CHP method. For this purpose, 5wt.%Cu powders were mixed with Al powders so as to form the liquid phase in the composites at temperatures over 548℃ as seen from Al-Cu phase diagram in Fig. 3.

This liquid phase fills the pores inside the composite and effects the consolidation. The consolidation is further enhanced by isostatic action of

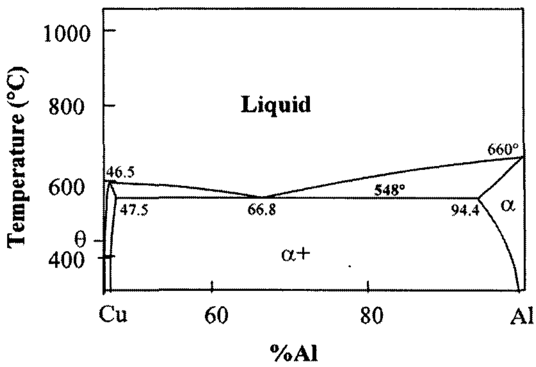

Fig. 3 Al-Cu phase diagram

the compressive stress on the compact inside the die. Additionally, diffusion rates increased with liquid phase and densification is enhanced by good wetting between liquid and solid components of the alloy system. On the other hand, during the hot pressing the time of contact between reinforcement and liquid phase is relatively short. Therefore, the reaction with oxygen is limited or blocked at the interface. As a result, the effect of the liquid phase in this technique becomes very important.

The SEM micrographs of Al-10B_4C and Al-30B_4C composites are shown in Fig. 4(a)-(b), respectively. The distribution of B_4C ceramic particles in composites is generally uniform. Fig. 4 (c) shows a higher magnification of B_4C particles, indicating a good bonding interface between the matrix and B_4C without evidence of pores.

These SEM results indicate the absence of pores in both interfaces and in the matrix, which also indicate that a good bonding between the matrix and ceramic particulate has obtained by using CHP method. On the other hand, our previous studies showed that several difficulties were encountered in hot pressing composites with reinforcing particle content above 30vol.%. A hot pressing temperature of 600℃ was not enough to consolidate either Al-40vol.%SiC or Al-40vol.% TiC composites. Even in successfully hot pressed Al-40vol.%SiC specimens, residual pores could be observed (Bedir, 2001).

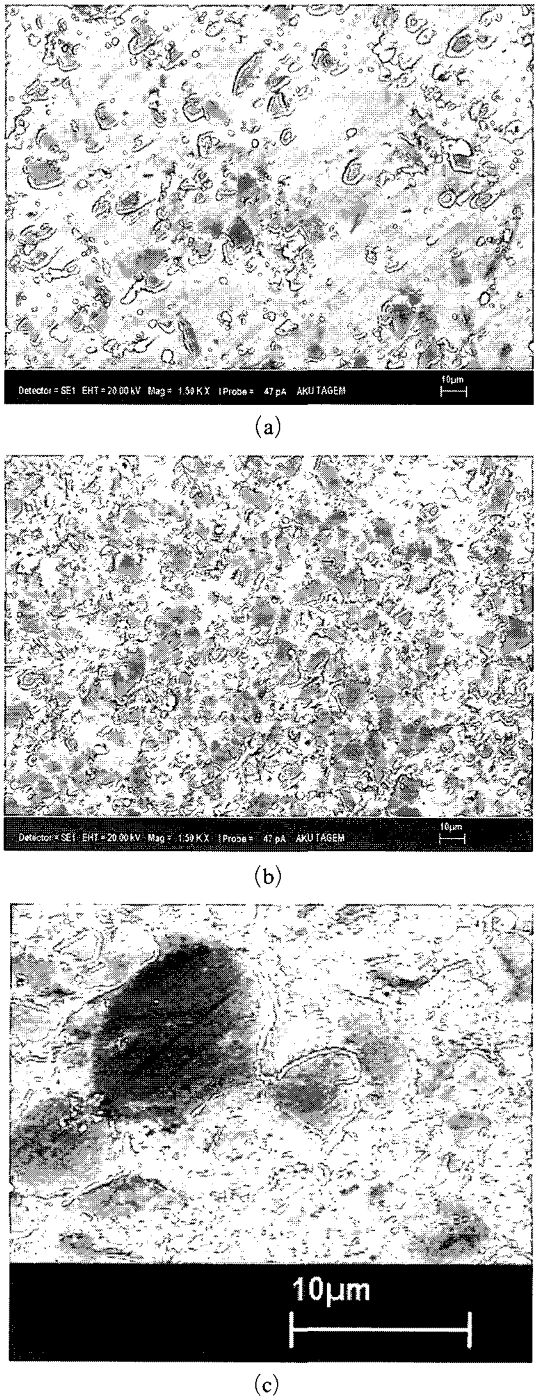

Fig. 4 Shows SEM micrographs of the Al-B_4C composites. In general, there appears to be a reasonably uniform distribution of the reinforcements

3.2 Age-hardening behavior

Al matrix composites are traditionally prepared from a pre-alloyed Al powder, in which the alloying elements are balanced to yield optimum ageing response. In current study, the age-hardening responses of the produced composites are investigated. The objective was to see the level of hardness attained in the hot pressed Al-Cu matrix alloy and its reinforced counterparts.

The effect of ageing time on the Brinell hardness of the Al-B_4C composites and matrix alloy are shown in Fig. 5 which gives the comparison of age-hardening responses for the composites and matrix alloy aged at 180 C. The hardness curve reveals a gradually continuous increase both in the composite and matrix alloy. The hardness of the Al-Cu matrix was increased from approximately 70 HB to 107 HB in 8 hrs (Fig. 5). No further change in hardness was observed, when the ageing period was extended up to 24 hrs. In all cases, the hardness of the reinforced composites was higher than the matrix alloy as expected.

The Al-30B_4C composite yielded the highest hardness value of 190 HB, upon ageing 8 hrs or more. As far as hardening behavior of the composites is concerned, particle addition in the matrix alloy increases the strain energy in the periphery of the particles in the matrix and these tendencies may be due to the formation of the dislocation at the boundary of the ceramic particles by the difference in the thermo-expansion coefficient between the matrix and ceramic particles during solution treatment and quenching since a lot of dislocations generate in the main matrix/particle interface (Kim, 2003; Salvador,

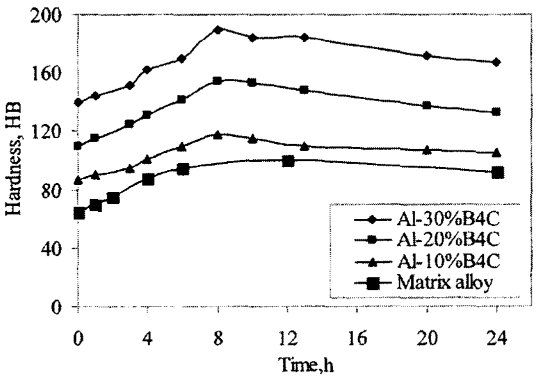

Fig. 5 Age-hardening behaviors of Al-composites at 180 C

2003; Das, 1996). Thus, dislocations cause the hardness increase in composite as well as residual stress increase because of acting as non-uniform nucleation sites in the interface following the age treatment. It is thought that the higher the amount of the ceramic particles in the matrix, the higher the density of the dislocation, and as a result, the higher the hardness of the composite.

In view of these results, it can be stated that a CHP of Al-B_4C composites can be effective method for the composites containing high volume fractions of reinforcements. Also, hot pressing method under nitrogen atmosphere; instead of expensive vacuum pressing method have another advantage for processing of cheaper composites, where mechanical properties are not so critical. However, it must be noted that the proper selection of the reinforcing phase seems to be critical to obtain sound specimens with the hot pressing conditions as described in this study.

3.3 Flexural Strength changes of composites and their fracture surfaces analysis

After age hardening process, it was approved that the hardness of composites has increased. Then three-point bending tests are applied on the specimens. As known, adding the ceramic particles in the composite, especially at high proportions makes microstructure brittle. For this reason, it is difficult or even impossible to prepare tensile bar specimens from these brittle composites to determine mechanical properties under tensile tests. Therefore, the most suitable test method for such brittle materials would be selected three-point bending tests. Previously, it was shown that hot pressed specimens had the highest hardness values within ageing 8 hours. For this reason, bending tests were applied on the specimens that aged 8 hours at 180℃.

Figure 6 shows the typical maximum flexural strength of different composites obtained from bending test. For single-layered composites, adding reinforcement to the composite influences on their mechanical behavior: the maximum flexural strength at no fraction of reinforcement; the minimum flexural strength at reinforcement 30Vol. percentage; drastically reduced flexural strength.

These results can be understood as a degree of the ductility of the composites. Since, the strength changes of the composites can be explained by internal plastic deformation occurring inside composite microstructure. During the cooling, internal plastic deformation arise due to the difference of thermal expansion coefficient between base alloy and reinforcements. Most equivalent plastic deformations were concentrated along the interface and propagated from the interface to base alloy. The distribution of stresses are compressive in base alloy region but high tensile around the reinforcements. This tensile stresses around the reinforcements are very high. On the other hand, this residual compressive stress plays a role in enhancing the strength of composite exposed tensile stresses under bending load. However, residual tensile stress around the interface can act as an initial defect, which causes debonding or early state fracture (Kang, 2005). Residual tensile stress around the interface increases with increasing particle content in the composite (Fig. 5). It can be conclude that bending result can be influenced by the constituent of the composite. However, it is believed that actual properties change is not only depended on the constituent of the composite (Choi, 2002) but also the microstructural variation and failure characteristics of composites.

For layered composites, bending results (Fig. 6) can be explained by residual stress distribution in the fracture surface. The residual stress in the layered composites is more complex phenomenon and much higher than that of the single layer composite. During cooling, the difference in deformation, owing to the different thermal expansion factor of the layers, is accommodated by creep as long as the temperature is high enough. Below a certain temperature, that will be called the joining temperature, the different components become bonded together and internal stresses appear (Lugovya, 1999). For each layers, total deformation after cooling is the sum of an elastic component and of a thermal component (Chartier, 1995).

In the view of this explanation, if layers are separated, Al-$10B_4C$ layer contracts more than Al-$30B_4C$ layer during cooling. However, in case of two-layered position in Fig. 7(a), the contraction of upper layer Al-$10B_4C$ is constrained by lower layer Al-$30B_4C$. This leads to a residual tensile stress occurred in upper layer, and a residual compressive stress in lower layer (Fig. 7 (a)). This chances stress distribution in the fracture area under bending condition and increases flexural strength of layered beam compared to equivalent Al-$30B_4C$ single layer. In the second case, the position of layers is changed. Al-$30B_4C$ is on the top position. In this case, as shown in Fig. 7(b), residual stress distribution affect flexural strength of the layered beam in decreasing if compared equivalent Al-$10B_4C$ single layer. This is also confirmed with the result obtained from bending as given in Fig. 6. In this stage, additionally, interface bonding affecting the layers, is

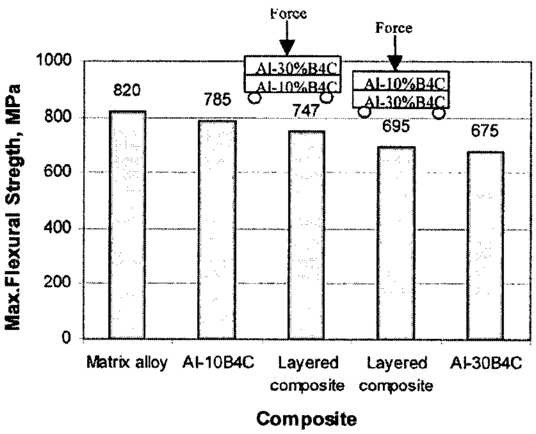

Fig. 6 Three-point bending test results of single layered and two layered composites

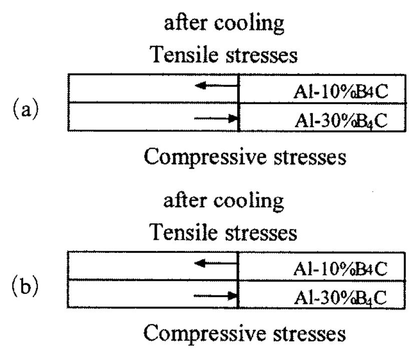

Fig. 7 Schematic illustration of residual stress distribution in the fracture surface

very important (Gursoy, 2005) and completely depends on composite type and production technique. Bending results are shown that a good interface bonding between the layers and between matrix and ceramic particulates were obtained by using the CHP method as can be seen in Fig. 8(c) and (d).

Figure 8 shows the corresponding SEM photographs of the fracture surfaces for each layer and transition region between layers (Fig. 8(d)). It shows dimple structures on the fracture surface of two types of the materials. The reinforcing particles are distributed uniformly in the dimples, and interface bond is well between the powders and matrix.

The formation of the dimples is the typical process for ductile crack propagation. It is well known that the failure in composite occurs by ductile fracture of the matrix between reinforcement particles. The particles, however, fail in brittle manner. Large dimples on the fracture surface indicate considerable void growth and plastic deformations before failure (Fig. 8(a)). Smaller dimples, on the other hand, indicate a fracture with less void growth and plastic deformation (Fig. 8(b)) (Rabiei, 2000).

The fracture mechanism of the composites can be depicted into three stages : void nucleation either by particle fracture or decohesion of the particle/matrix interface, growth and finally coalescence of the voids to give total failure of the material (Da Silva, 2005). The higher magnification of the void leaved behind by B_4C particle in the matrix during failure is shown Fig. 8(c).

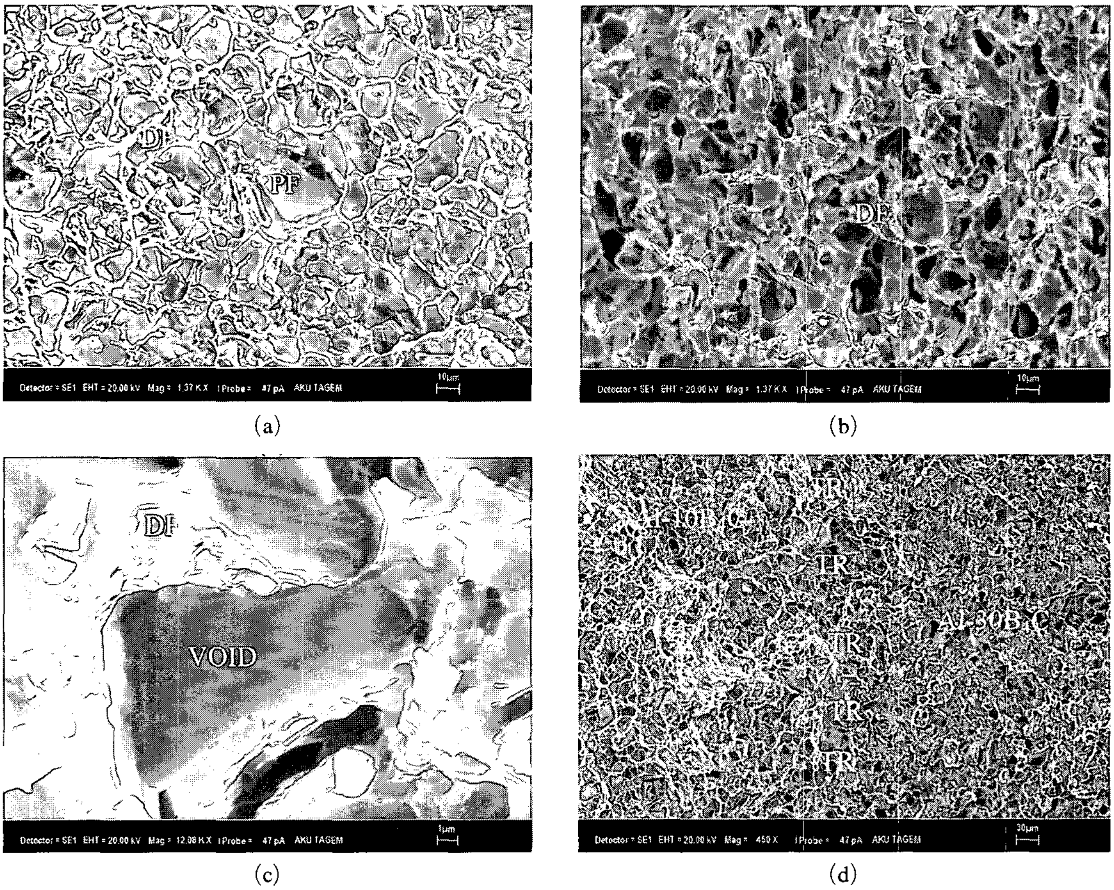

Fig. 8 SEM micrograph taken from the fracture surface of the bending specimens tested at room temperature. DF : ductile fracture. PF : particle fracture. TR : transition region between layers

4. Conclusions

In this study, an alternative producing method is proposed. Hot pressing of powders with a little liquid phase is a simple and at least complicated powder consolidation method, which is used to fabricate Al-MMCs with homogeneously dispersed particles without porous structures. The ageing behavior of the composites was found to be influenced by the reinforced materials, i.e. higher hardness values were reached in 8 hrs for the composites than that for the matrix alloy. The thermal mismatch between the metal matrix and the reinforcement increases the dislocation density of the matrix. Thus, dislocations cause the composite to increase in hardness.

The mechanical properties of the specimens were determined by three-point bending test. For single-layered composites, adding the reinforced particulate to the composites influenced on their mechanical behavior as a result reduced failure strength. For two-layered composites, the residual stress in the layered composites is more complex phenomenon and much higher than that of the single-layered composite. If layered beam of which upper layer has less reinforcement content in matrix than lower layer, flexural strength of the beam increased according to equivalent down counterpart due to the distribution of the residual stresses in the cross-section on the loading plane.

The fracture surface of the composite material consisted of voids which formed by the strain localization at sharp corners of B_4C particles. These voids were then coalesced during bending, resulting in the formation of dimple appearance at the fracture surface. The fracture mechanism can be evaluated into the following stages : nucleation of the voids at the B_4C particles (particle fracture), growth of these voids followed by ductile failure of the matrix by coalescence of micro voids and finally coalescence of the voids to give total failure of the material.

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