Mechanical Properties and Fracture Process of SiC_w/Mg Composites Produced by Squeeze Casting and Extrusion

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Mechanical properties of SiC whisker reinforced magnesium-matrix composites with random and unidirectional alignment of whiskers produced by squeeze casting and hot extrusion were investigated under room and elevated temperature atmospheres. The use of AE42 alloy instead of pure magnesium as a composite matrix contributes to an improvement of mechanical properties, especially proof stress of the composite. The extruded composites have more excellent mechanical properties such as tensile strength, proof stress and elongation than those of the cast composites at room temperature. At elevated temperatures, however, a remarkable decrease of tensile strength and proof stress, and an increase of elongation take place for the extruded composites than for the cast composites. The fracture occurs by cracks which propagate from the whisker/matrix interface debonded at whisker ends to matrix at temperatures lower than 473 K. On the other hand, the fracture takes place by cracks propagating through the matrix/whisker interface at above 473 K.

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I. Introduction

Magnesium alloys with low density have been noticed quite recently to solve the problem of environment pollution and energy in all industrial fields. However, magnesium alloys have been not used for high performance applications such as in the aircraft and automotive industries due to low mechanical properties at room and elevated temperatures. Therefore, magnesium alloys-based metal matrix composites (MMC) with various reinforcements have been produced by powder metallurgy⁽¹⁾⁽²⁾ and squeeze casting⁽³⁾⁻⁽⁶⁾, to obtain the higher specific mechanical properties necessary to achieve better performance.

In general, the squeeze casting is being used for producing composites because of the several merits that are possible to produce goods in short time and cheap cost, to eliminate shrinkage and gas porosities and to reduce a damage of reinforcements due to the short reaction time between matrix melt and reinforcements. It is known that the fabrication of excellent MMC by squeeze casting depends upon the quality of preforms, control of the thermal conditions and mechanical conditions such as pressure, plunger speed, etc., during casting⁽⁷⁾⁻⁽⁹⁾.

The appearance and shape of the matrix/reinforcements interface are very important factors to analyse the fracture process of MMC under the tensile test, because the increase of strength and elastic modulus can be obtained through successful transfer of the load from the ductile matrix to the strong reinforcements⁽¹⁰⁾.

The aim of this work is to study in detail the effect of SiC whisker reinforcements on the mechanical properties

and to clarify the fracture process under tensile-tests at room and elevated temperatures in the SiC whisker reinforced magnesium matrix composites produced by squeeze casting and hot extrusion.

II. Experimental Procedure

The matrix materials used were pure magnesium with the purity of 99.9 mass% and a Mg-Al-Rare Earth alloy AE42 having high creep-resistance⁽¹¹⁾⁽¹²⁾. The chemical composition of the AE42 alloy is presented in Table 1. Matrix materials were melted under the mixed gas atmosphere of SF₆(25%) and CO₂(bal.) at 1093 K, cast into a mold set with a preform and then reinforced with 30% by volume of SiC whiskers. The SiC whiskers were typically 0.5 μm in diameter and 15 μm in length. The SiC whisker preforms with dimensions of φ 55 mm × 50 mm were preheated in a furnace under Ar gas atmosphere at 1223 K for 2 h. The melt was poured into the preheated mold where a preform was set. Composites were produced by squeeze casting using the pressure of 100 MPa, the plunger speed of 23 mm/s and the mold temperature of 573 K. Figure 1 shows the scheme of the squeeze casting method used for the fabrication of composites. Composites thus obtained were treated as cast composites. A half of the cast composites were hot-extruded the conditions of the extrusion ratio of 10:1 and the extrusion

Table 1 Chemical composition of AE42 alloy.

(mass%)

Element	Al	Ce	La	Pr	Nd	Mn	Cd	Fe	Mg
Amount	4.03	1.26	0.63	0.11	0.38	0.26	0.01	0.002	bal.

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Fig. 1 Schematic drawing of a squeeze casting method for the fabrication of SiC_w/Mg, AE42 alloy composites.

speed of 11 mm/s, and they were treated as extruded the composites. The homogenization treatment for the unreinforced AE42 materials and the reinforced AE42 composites was performed at 683 K for 24 h. Tensile test specimens with a diameter of 3.6 mm and a gauge length of 14 mm were machined from both the unreinforced materials and composites. The densities of cast and extruded composites were measured to confirm the change in volume fraction of SiC whiskers by squeeze casting and hot extrusion, and they are shown in Table 2. Pure magnesium and the AE42 matrices composites were heat-treated for 1 h at 773 and 673 K, respectively and tensile-tested under condition of strain rate of approximately 6×10^{-4} /s at room and elevated temperatures (423, 473, 573 K).

The sections perpendicular to the fracture surfaces of the unreinforced AE42 materials were observed by the optical microscope. In order to make clear the fracture process of composites, the whisker/matrix interfaces and the surface of the whiskers after tensile test were examined by a field emission scanning electron microscope (FESEM). And a transmission electron microscope (TEM) and an electron probe micro analyser (EPMA) were used to analyse reaction products.

Table 2 Change of volume fraction of SiC whiskers by squeeze casting and hot extrusion.

	Volume fraction (%)		
	Preform	Cast	Extruded
SiC _w /Mg	30	35.6	39.5
SiC _w /AE42	30	36.2	39.0

III. Results and Discussion

1. Microstructures of composites by squeeze casting and extrusion

Optical micrographs of AE42 alloy produced by squeeze casting and hot extrusion are shown in Fig. 2. The cast and extruded AE42 matrices contain the crystallized compounds identified as Al-RE binary compound by EPMA analysis. Recently, Sakkinen⁽¹³⁾ reported that the compounds in AE42 alloy were Al₄RE. After homogenization treatment at 683 K for 24 h, those crystallized compounds did not disappear.

The structures of the cast and extruded SiC_w/AE42 alloy composites are shown in Fig. 3(a) and (b), respectively. The structures of a cast composite with a random alignment of whiskers and an extruded composite with an unidirectional alignment of whiskers show hexagonal and triangular shape whiskers broken during casting and hot extrusion. However, the composites thus produced were regarded as sound composites in spite of the existence of some broken whiskers.

Figure 4 shows the TEM images of the extruded SiC whisker reinforced Mg composite heat-treated at 773 K for 1 h. In the as-heat treated state, the composite includes dislocations which are generated on cooling from the fabrication and heat treatment temperatures, and good bonded interfaces between the matrix and whiskers. Reaction products with a spherical shape were analysed as MgO which had formed on the whisker surface at the matrix/SiC interface.

Fig. 2 Optical micrographs of (a) cast and (b) extruded AE42 alloys.

Fig. 3 SEM images showing SiC whiskers distributed (a) randomly and (b) unidirectionally in SiC_w/AE42 alloy composites.

Fig. 4. TEM micrographs and diffraction pattern showing dislocations generated by thermal stress and MgO after heat treatment at 773 K for 1 h.

2. Influence of matrix and alignment of whiskers on tensile properties of composites

Figure 5 shows the elastic modulus, the 0.2% proof stress and the tensile strength of unreinforced matrices and reinforced composites at room temperature. It is obvious that there are increases in the elastic modulus, the 0.2% proof stress and the tensile strength by compositing pure magnesium and AE42 alloy matrices with SiC whiskers. The elastic modulus and the 0.2% proof stress of extruded matrices and composites were superior to those of cast matrices and composites because of the refined grain structure, unidirectional alignment of whiskers and improvement of bonding of matrix/whisker interfaces by extrusion. The change of matrix alloy from pure magnesium to AE42 alloy contributes to a small increase of the elastic modulus, and a large increase of the 0.2% proof stress. The 0.2% proof stress of cast SiC_w/AE42 composite was not obtained and the tensile

strength of cast SiC_w/AE42 composite was low. It results from that the AE42 matrix alloy contains a lot of large crystal compound which play a role in the origin of fracture under the tensile test. And, it is also obvious from the result that the strengthes of the cast AE42 alloy matrix and SiC_w/AE42 composite increase markedly by extrusion because the large crystal compound is refined by extrusion, but the increase of strengthes by extrusion in pure magnesium and SiC_w/Mg composite without any large compound is small. On the other hand, the elongations of cast and extruded composites were very small, approximately less than 0.5%, and there was no influence of the hot extrusion and the change of matrix on the elongation of composites at room temperature. As a result of compositing the AE42 alloy and pure magnesium with SiC whiskers, the magnesium matrix composites with the specific elastic modulus and tensile strength higher than those of the conventional materials can be obtained as shown in Table 3.

Fig. 5 Elastic modulus, 0.2% proof stress and tensile strength of unreinforced matrices and reinforced composites at room temperature.

Table 3 Specific elastic modulus and specific tensile strength of composites compared with some typical high strength metallic materials.

Material	Specific elastic modulus (E/ρ)	Specific tensile strength (σ/ρ)
Cast		
Mg	16.4	63.2
AZ91 ⁽¹⁴⁾	19.4	89.6
AE42	22.8	93.5
SiC _w /Mg	36.8	185.7
SiC _w /AZ91(Vf30) ⁽¹⁴⁾	32.6	168.5
SiC _w /AE42	37.8	157.7
Extruded		
Mg	22.3	72.8
AZ91 ⁽¹⁴⁾	26.2	163.8
AE42	23.1	144.8
SiC _w /Mg	48.5	204.4
SiC _w /AZ91(Vf30)-T6 ⁽¹⁴⁾	52.5	173.3
SiC _w /AZ31(Vf30) ⁽¹⁵⁾	44.3	174
SiC _w /AE42	48.5	237.9
A7075-T6	25.4	207
Ti-6Al-4V	25.8	262
Maraging steel (18Ni350)	23.3	300

In the pure magnesium materials and composites, the change in tensile strength and elongation with increasing temperature is shown in Fig. 6. The tensile strengths of extruded and cast composites decline remarkably with increasing temperature, and there is almost no difference in strength of extruded and cast composites at 573 K. However, the effect of compositing with SiC whiskers on the tensile strength at elevated temperatures is marked, that is, the tensile strength of composites at 573 K is about 110 MPa which is about seven times as large as that of the matrix materials. The remarkable lowering in the tensile strength with increasing temperature is also ob-

Fig. 6 Changes in tensile strength and elongation with increasing temperature in unreinforced Mg materials and SiC_w/Mg composites.

Fig. 7 Changes in tensile strength and elongation with increasing temperature in AE42 alloy materials and SiC_w/AE42 composites.

served in AE42 composites as shown in Fig. 7. The reasons why remarkable lowering of tensile strength occurs are as follows. Matrix/whisker bonding gets worse, and the deformation of matrix such as slip and twinning which play important roles in deforming at various tem-

Fig. 8 Optical micrographs of the surfaces perpendicular to fracture surfaces of unreinforced AE42 alloy materials tensile-tested at R.T., 423 and 573 K.

peratures⁽¹⁰⁾ occurs easily, and the recovery of dislocations and stress by recrystallization in the extruded composite occurs fast with increasing temperature. For the AE42 composites, the decrease of the tensile strength with increasing temperature was modest in comparison with the pure magnesium composites. On the other hand, the change in 0.2% proof stresses of matrix materials and composites at elevated temperatures appeared the same tendency as that in the tensile strength.

Figures 6 and 7 also show the influence of temperature on elongations of the pure magnesium unreinforced matrix and reinforced composite and AE42 unreinforced matrix and reinforced composite respectively. The elongations of the extruded ones are higher than those of the cast ones because of the fine grain structures and good bonding of the matrix/whisker interfaces. However, the refined grain structures by hot extrusion were not observed in pure magnesium, therefore, the elongations in the cast and extruded magnesium materials are hardly different at elevated temperatures. By the way, the extruded AE42 alloy material reveals elongation much higher

than the cast one. In the case of the pure magnesium composite, the elongation at temperatures higher than 423 K decreased due to the reason that the generation and connection of voids proceeded fast during the tensile test. However, the elongation of the extruded SiC_w/AE42 composite increase with increasing temperature, this results from improvement of the bonding between the matrix and whiskers due to change of matrix from pure magnesium to AE42 alloy material. That is, it means that the generation and connection of voids at elevated temperatures under the tensile test decrease by using the matrix which bonds well with SiC whisker.

3. Fractographs, morphology of whisker surface and whisker/matrix interface reaction

The optical microscope photographs of the surfaces perpendicular to fracture surface of unreinforced AE42 alloy materials are shown in Fig. 8. The generation of voids increases with increasing temperature, especially a lot of voids generate during the tensile test at 573 K. Therefore, the fracture of AE42 alloy materials at elevat-

Fig. 9 FE-SEM images showing fracture surfaces of (a) cast and (b) extruded SiC_w/AE42 composites tensile-tested at R.T., 423, 473 and 573 K.

ed temperatures occurs by the connection of generated voids.

FE-SEM photographs of fracture surfaces of the AE42 alloy cast and extruded composites at various temperatures are shown in Fig. 9. The fracture surface of the composite changes into a ductile surface with increasing temperature. The fracture surface of the cast composite reveals the whiskers perpendicular to the tensile direction, but the pulled-out whiskers are not observed. This indicates excellent bonding between the matrix and whiskers at room temperature. There is no difference in

the fracture surfaces at room temperature and 423 K. At temperatures higher than 423 K, the deformed matrix and the pulled-out whiskers from the matrix are observed. The change of the fracture surfaces with increasing temperature corresponds to the decrease of tensile strength. On the other hand, the fracture surfaces of the extruded composites show a cleavage morphology. And broken whiskers are also observed, which might be broken under the composition fabrication and the tensile test. The change of fracture surfaces of the extruded composites with increasing temperature is similar to that of

Fig. 10 FE-SEM images showing surfaces of whiskers in extruded SiC_w/Mg composites fractured at P.T., 423 and 573 K.

the cast composites, the deformed matrix alloy is observed at 473 and 573 K. However, the bumpiness of the fracture surface of the extruded composites is small in comparison with that of the cast composite, therefore, the complete pulled-out whiskers are almost no observed.

The surfaces of whiskers in the fractured pure magnesium composites are shown in Fig. 10. The whiskers perpendicular to the tensile direction are well bonding with the matrix at temperatures lower than 473 K. At higher temperatures, the matrix is perfectly separated from the surface of whiskers, the spherical reaction products MgO are observed on the surface of whiskers. It was reported that Mg₂Si and MgO phases were formed by a chemical reaction between the magnesium alloy materials and the SiO₂ coating of reinforcements⁽¹⁶⁾. When pure magnesium is used for matrix materials of composites, Mg reacts with SiO₂ and then MgO forms at the matrix/SiC whisker interfaces as shown in Fig. 4. There are basically two possible reactions for the formation of MgO at the interface.

And in the case of the magnesium alloy materials containing aluminum, the following two reactions with SiC and SiO₂ are considerable⁽¹⁷⁾⁻⁽²¹⁾.

In the case of reaction eq. (2), (3) and (4), Mg₂Si, Al₄C₃ and Al₂O₃ are possible reaction products⁽²²⁾. In this study, however, Al₄C₃ and Al₂O₃ have not been observed at the matrix/whisker interfaces. On the other hand, skeleton-shape reaction products were observed in the melt pushed-out from the SiC preform through the filter

by squeeze casting (the area A at Fig. 1), and they were identified as Mg₂Si by EPMA. And also, rod-shaped Al-RE crystallized compounds which are formed during the infiltration are shown in Fig. 11. Generally, the chemical reaction, diffusion of atoms from one component to the other and good wettability of a liquid phase can give rise to a mechanical and physical interaction at the matrix/whisker interfaces, allowing the development of a strong bonding. The separation between the matrix and whiskers and the separation of reaction products from the whisker surface are observed in the extruded composites at 573 K. This means that both bondings between whiskers and reaction products and between whiskers and the matrix get worse with increasing temperature. However, the effect of bonding between whiskers and reaction products on the mechanical properties of composites is not manifested yet. Therefore, we consider that the reaction products do not affect the mechanical properties when they are extremely small in size and amount. After all, the separation of the matrix from the whisker surfaces in the composites with increasing testing temperature is one of the important factors which deteriorate the mechanical properties of the SiC whisker reinforced pure magnesium and the AE42 alloy based composites produced by squeeze casting and extrusion.

4. Representation of fracture process

From the observation of fracture and whiskers surfaces after the fracture of composites, the fracture processes around the whisker ends are summarized in Fig. 12. Cracks can be caused on the whisker edges by the thermal shock which occurs during solidification and/or quenching due to the difference between the coefficients of thermal expansion (CTE) of whiskers and the matrix and the mechanical shock which occurs during casting and hot-extruding. However, it is very difficult to distinguish cracks by thermal shock from cracks by mechanical shock. The

Fig. 11 EPMA showing Mg₂Si with a skeleton shape and compounds with a rod shape formed in AE42 alloy melt (part A in Fig. 1) which was pushed-out from SiC whisker preform during squeeze casting in SiC_w/AE42 alloy composite.

cracks on the whisker edges propagate into whiskers and the matrix under the tensile test as shown in the first fracture process. On the other hand, the whisker/matrix interface A in the figure is well bonded, and the interface B is badly bonded because most of the whiskers are broken by mechanical shock during the squeeze casting and hot extrusion. Cracks are also formed from the debonded interface between whisker and matrix, which occur easily at the interface B during tensile deformation. The tensile stress concentration at the tip of tensile cracks propagates the tensile cracks into the matrix, as the second fracture process shown in Fig. 12. On the other hand, the shear yielding may proceed in the matrix near the interface A due to shear stress concentration around the whisker ends and cause the shear cracking at the edge of whiskers finally⁽²³⁾. However, the strong bonding between the matrix and whiskers prevents the crack from propagating, therefore, the crack propagates into the

matrix. This is the third fracture process in Fig. 12. When the second and third fracture processes occur continuously, the propagated cracks reach the other whisker/matrix interface and proceed to the tip of whiskers through the whisker/matrix interface by the concentration of stress. However, if the bonding of whisker/matrix is strong, the cracks might be not proceed. So, the cracks may proceed by breaking the whisker in the 4th fracture process. Figure 13 shows typical examples of tensile fracture surface which might be fractured through the process 1 ~ 4. Eventually, the fracture of the SiC whisker reinforced magnesium-matrix composites occurs certainly by the continuous and simultaneous proceeding of the fracture processes 1 ~ 4 in the long run. Kwon⁽²⁴⁾ represented that if discontinuous whiskers are aligned ideally in an extruded composite, the fracture occurs by the propagation of cracks. It is considered that the second fracture process might occur easily at low temperatures owing to maintain-

Fig. 12 Schematic representation of the fracture process around the whisker ends.

Fig. 13 FE-SEM images showing typical examples of tensile fracture surfaces which might be fractured through the processes 1, 2, 3 and 4.

ing good bonding of the matrix/whisker interface A. At high temperatures, the third fracture process takes place preferentially against another fracture process due to the separation of the matrix/whisker interface A resulting from a change for the worse of the bonding of matrix and whisker. And the fracture of the extruded composite compared with the cast one might occur by the 1st and 2nd processes at various temperatures because of the good bonding of the whisker/matrix interfaces A and the occurrence of many damaged whiskers by extrusion. Actually, the distribution, alignment and volume fraction of whiskers play significant roles in determining the mechanism of the fracture of the SiC whisker-reinforced magnesium matrix composites.

IV. Conclusion

The changes of the matrix for the composite from pure magnesium to AE42 alloy and the alignment of whiskers are valid to improve the mechanical properties of the SiC whisker reinforced magnesium-matrix composites. Mechanical properties of the extruded composites with a unidirectional alignment of whiskers are superior to those of the cast composites with a random alignment of whiskers at room temperature, that is, UTS of the extruded AE42 composite is 548 MPa, which is 200 MPa higher than that of the cast composite.

The lowering in tensile strength of the extruded composites is faster than that of the cast ones with increasing temperature. The strengths of the extruded composites increase above 100 MPa at temperatures lower than 473 K by using the AE42 alloy instead of pure magnesium. The SiC_w/Mg and SiC_w/AE42 alloy composites have higher tensile strength above 100 MPa at 573 K than matrix materials.

The reaction product, MgO, forms at the matrix/whisker interface, and the other product, Mg₂Si, also exists at the part pushed-out from the SiC whisker preform.

The deterioration of bonding between the matrix and whiskers with increasing temperature is one of the important factors to cause the decrease in tensile strength and proof stress at elevated temperatures. And four fracture processes under the tensile test are proposed. The first fracture process shows the fracture due to cracks generated on the whisker edges by thermal and mechanical shocks. The second process is the fracture which takes place by cracks propagating from the whisker/matrix interface debonded at the whisker ends to the matrix. The third fracture process is due to the cracks propagating through the whisker/matrix interface. The cracks proceeding by breaking the whisker also give rise

to the fracture, the fourth fracture process. In the SiC whisker reinforced magnesium-matrix composites produced by squeeze casting and extrusion, the second fracture process plays a significant role in fracture at temperatures lower than 473 K. On the other hand, the third fracture process is a dominative fracture process at above 473 K. However, the fracture of composites resulted from the continuous and simultaneous progress of the proposed fracture processes.

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