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ARTICLE

Effects of SiCp Content on the Microstructure and Mechanical Properties of SiCp/Mg-5Al-2Ca Composites

Chang Hai[1], Wang Cuiju[2], Deng Kunkun[2], Nie Kaibo[2], Su Kun[2], Liao Ling[2], Wang Hongxia[2], Tong Libo [3]

[1] National Center for Materials Service Safety, University of Science and Technology Beijing, Beijing 100083, China; [2] Shanxi Key Laboratory of Advanced Magnesium-based Materials, Taiyuan University of Technology, Taiyuan 030024, China; [3]State Key Laboratory of Rare Earth Resources Utilization, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun 130022, China

Abstract: The Mg-5Al-2Ca alloys reinforced by 2vol%, 5vol% and 10vol% 5 μm SiCp were fabricated by stir casting. The cast composites were subject to hot extrusion. In the cast composites, addition of SiCp disturbs the network distribution of Al_2Ca phase along grain boundaries. After extrusion, grains of the matrix are refined further and the Al_2Ca phase break into particulates. Moreover, the Al_2Ca phase and grain size can be further refined by the addition of SiCp. Compared with Mg-5Al-2Ca alloy, the addition of SiCp introduces obvious improvement of tensile yield strength (TYS) and work hardening rate, however, at the expense of elongation. Unlike the TYS, the ultimate tensile strength (UTS) of SiCp/Mg-5Al-2Ca composite does not change monotonously with increasing SiCp's content, and the highest UTS appears when the SiCp's volume fraction is 5%. The interfacial debonding between SiCp and Al_2Ca phase and cracked particles are considered as the main crack source for SiCp/Mg-5Al-2Ca composite.

Key words: magnesium matrix composite; SiCp; reinforcement phase; microstructure; mechanical property

The addition of Ca, as a low cost and density (1.55 g/cm^3) element, into Mg-Al alloys can form stable intermetallic compounds, such as (Mg, Al)$_2$Ca, Al_2Ca and Mg_2Ca[1-4]. Due to the thermally stable precipitated phases above, Mg-Al-Ca system alloys possess high hardness, good creep resistance and castability, better oxidation resistance as well as high ignition temperature, etc.[2]. However, the precipitation of (Mg, Al)$_2$Ca, Al_2Ca or Mg_2Ca phase along grain boundaries usually deteriorated the mechanical properties of as-cast Mg-Al-Ca alloys[3-5]. On Wang et al.'s research of SiCp/Mg-Zn-Ca composite, the distribution of precipitated phase can be modified by addition of the micron SiCp[6]. Nevertheless, the effect of SiCp on precipitating behavior of intermetallic phases still needs further investigation.

As it is known the casting defects which cannot be fully avoided during solidification can be eliminated by secondary deformation process[7-9]. It has been shown that the application of hot extrusion could result in uniform particle distribution and refined grain in the particle reinforced magnesium matrix composite, leading to obviously improved mechanical properties[10-13]. The author's previous work on Mg-Al-Ca alloy indicated that the Al_2Ca phase broke into particulates during extrusion and were distributed along extrusion direction afterwards[14]. Latest work of author's group demonstrated that the addition of 1vol% SiCp has obvious effects on Al_2Ca phase refinement and mechanical property improvement of Mg-Al-Ca alloy [15]. Nevertheless, the content of SiCp in previous work is rather low (only ~1vol%). The influence of SiCp's content on Al_2Ca phase and its combining effects on the microstructure and mechanical properties of Mg-Al-Ca alloy still needs further investigations.

In this study, the SiCp reinforced Mg-5Al-2Ca

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Corresponding author: Deng Kunkun, Ph. D., Professor, Shanxi Key Laboratory of Advanced Magnesium-based Materials, College of Materials Science and Engineering, Taiyuan University of Technology, Taiyuan 030024, P. R. China, Tel: 0086-10-62333860, E-mail: jamsdk@163.com

composites with different volume fractions of 2vol%, 5vol% and 10vol% were fabricated by stir casting and then subjected to hot extrusion. Precipitation behavior of the precipitated phases was revealed. Then, the interaction between precipitated phase and SiCp as well as their combining effects on Mg-5Al-2Ca matrix was discussed in detail.

1 Experiment

The Mg-5wt%Al-2wt%Ca was selected as matrix alloy and the SiCp with the average particle size of ~5 μm was selected as reinforcement. Three kinds of composites with different SiCp volume fractions (2vol%, 5vol% and 10vol%) were fabricated by stir casting. Firstly, the Mg–5Al–2Ca alloy was molten at 993 K and cooled to 873 K at which the matrix alloy is in semi-solid condition. Then, the SiCp preheated to 873 K was quickly added into the semi-solid matrix alloy. At last, the melt was heated up to 1023 K and poured into a preheated steel die for casting.

Cylindrical billets with 40 mm in diameter were machined from the cast composite ingots. The billets were extruded into round rods at 673 K with an extrusion ratio of 16:1. The ram speed was 3 mm/s and graphite-based mixture was used as a lubricant.

Microstructures observation were carried out by optical microscopy (OM) and scanning electron microscope (SEM) equipped with energy dispersive spectrometer (EDS). Specimens for OM and SEM were etched in acetic picral (2.1 g picric acid + 5 mL acetic acid + 5 mL distilled water + 35 mL alcohol) after mechanical polishing. The average grain size was measured using Image-pro Plus 6.0 software. The phase composition was tested by X-ray diffractometer (XRD). The tensile specimens were cut parallel to extrusion direction. At least three specimens for each sample were conducted on an Instron 5569 testing machine at a constant speed of 0.5 mm/min at room temperature. Fracture surfaces were observed by SEM.

2 Results and Discussion

2.1 Microstructure of the as-cast composite

Fig.1 shows the SEM microstructures of as-cast alloy and composites reinforced by 2vol%, 5vol% and 10vol% SiCp. Precipitated phases are distributed along grain boundaries as network in the as-cast Mg-5Al-2Ca alloy, as shown by Fig.1a and 1b. The composition of point "A" and "B" in Fig.1b according to EDS is mainly Mg, Al, and Ca elements. The atomic ratio of Al:Ca is 2.15:1 and 2.07:1, as shown in Table 1. Thus, the precipitated phase could be determined as Al_2Ca, as similar as previous work on Mg-5Al-2Ca alloy[14]. Fig.1c~1h reveal the morphology and distribution of Al_2Ca phase in SiCp reinforced composites. As the volume fraction of SiCp is 2%, a little amount of Al_2Ca phases could be found along the grain boundaries while majority of them present a granular form, as demonstrated in Fig.1c and 1d. With increasing of the SiCp volume fraction, the Al_2Ca network vanishes and gradually turns into granular form, as shown in Fig.1e, 1f. To identify SiCp and Al_2Ca phase clearly, detailed EDS mapping of the extruded 2vol%, 5vol% and 10vol% SiCp/Mg-5Al-2Ca composites are shown in Fig. 2. The detailed composition of the points "C", "D", "E", "F", "G" and "H" is also summarized in Table 1. It is demonstrated that, for all SiCp/Mg-5Al-2Ca composites, most granular Al_2Ca phase precipitates on the surface of SiCp during solidification and the Al_2Ca phase disperses homogenously in the cast composite.

2.2 Microstructure of the extruded composite

Fig. 3 shows the X-ray diffraction patterns of as-extruded

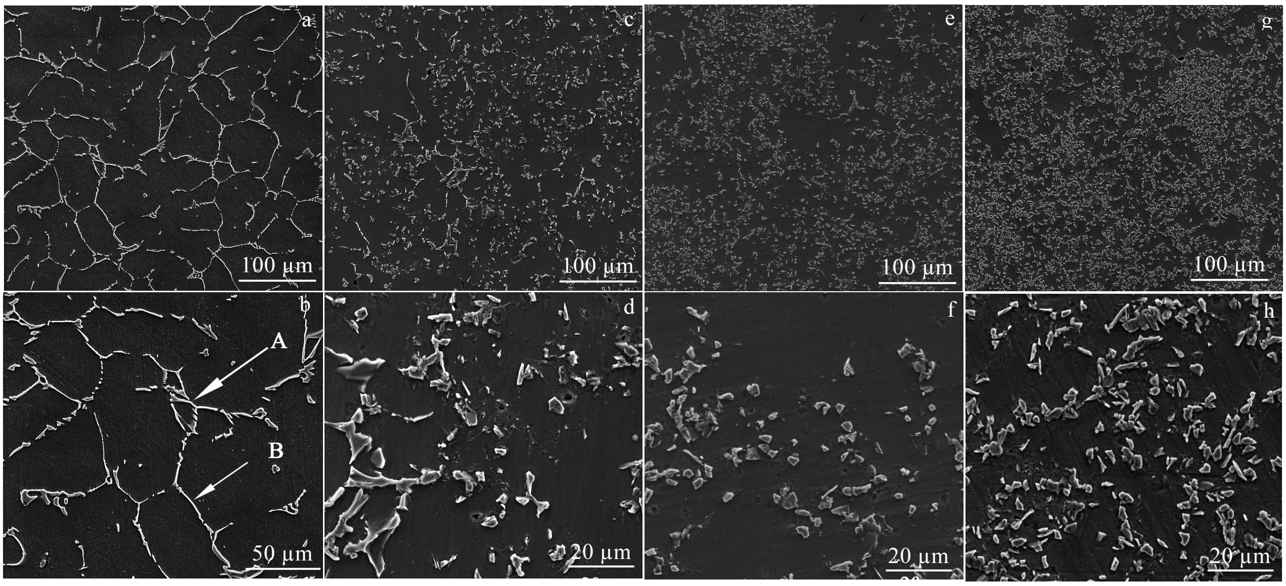

Fig.1 SEM images for the as-cast (a, b) alloy and composites with 2vol% (c, d), 5vol% (e, f), and 10vol% (g, h) SiCp

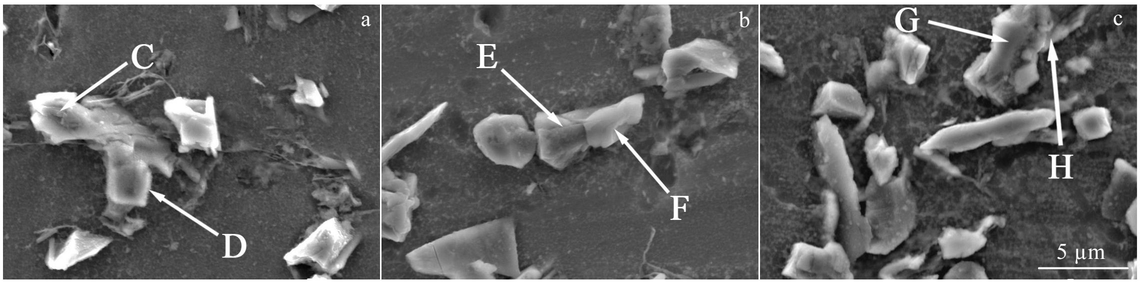

Fig.2 SEM images and EDS analysis positions of 2vol% (a), 5vol% (b), and 10vol% (c) as-cast SiCp/Mg-5Al-2Ca composites

Table 1 EDS results of the as-cast alloy and composites (at%)

Positions	Elements					Possible compounds
	Mg	Al	Ca	C	Si	
Fig.1b-A	17.0	56.6	26.3	—	—	Al_2Ca
Fig.1b-B	55.1	30.2	14.6	—	—	Al_2Ca
Fig.2a-C	3.5	0.7	0.3	37.0	58.6	SiC
Fig.2a-D	14.7	51.8	24.9	8.7	—	Al_2Ca
Fig.2b-E	1.1	0.2	0.1	58.3	40.3	SiC
Fig.2b-F	14.7	57.5	27.9	—	—	Al_2Ca
Fig.2c-G	1.0	0.2	0.1	56.9	42.0	SiC
Fig.2c-H	10.8	61.9	27.3	—	—	Al_2Ca

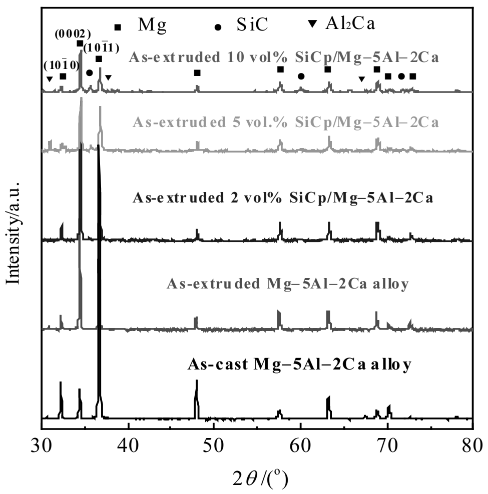

Fig.3 XRD patterns of as-extruded Mg-5Al-2Ca alloy and SiCp/Mg-5Al-2Ca composites

Mg-5Al-2Ca alloy and SiCp/Mg-5Al-2Ca composites. The Al_2Ca is the only precipitated phase in Mg-5Al-2Ca alloy and SiCp/Mg-5Al-2Ca composites. The higher relative intensity ratio of basal plane and pyramidal plane ratio of as-extruded Mg-5Al-2Ca alloy indicates that the strong basal plane texture forms during extrusion process, which is common in extruded Mg alloys[16].

The SEM microstructures of the as-extruded Mg-5Al-2Ca alloy and SiCp/Mg-5Al-2Ca composites are shown in Fig.4. For Mg-5Al-2Ca alloy, the large Al_2Ca phases distributed along grain boundaries (Fig.1a and 1b) break into particulates and the bands consisting of Al_2Ca phase are parallel to the extrusion direction, as shown in Fig.4a, and 4b. After the addition of 2vol% SiCp, the Al_2Ca phase bands become unclear, as shown in Fig.4c, and 4d. As the SiCp content increases from 5% to 10%, the Al_2Ca bands along extrusion direction are hard to be recognized, as shown in Fig.4e~4h. The average size of Al_2Ca in both Mg-5Al-2Ca alloy and SiCp/Mg-5Al-2Ca composites are summarized in Table 2 according to EDS analysis. The Al_2Ca phase in SiCp/Mg-5Al-2Ca composite is much finer than that in Mg-5Al-2Ca alloy. The average size of Al_2Ca particulates decreases gradually as the SiCp increases from 2vol% to 10vol% in the cast composites. After extrusion, the Al_2Ca phase is refined further so that the average size of Al_2Ca is reduced. Moreover, it is noticeable that the reduction of Al_2Ca size becomes less evident with increasing of the SiCp volume fraction during extrusion.

Fig.5 shows the optical microstructure of extruded Mg-5Al-2Ca alloy and SiCp/Mg-5Al-2Ca composites. Two kinds of grains could be observed in the extruded Mg-5Al-2Ca alloy, the fine equalized grains resulting from the dynamic recrystallization (DRX) and the deformed grains along extrusion direction, as shown in Fig.5a. The average grain size of the as-extruded Mg-5Al-2Ca alloy is

Table 2 Mean diameter of the second phase in Mg-5Al-2Ca alloy and SiCp/Mg-5Al-2Ca composites (μm)

Materials	As-cast	As-extruded
Mg-5Al-2Ca	4.66±3.66	1.17±0.61
2vol% SiCp/Mg-5Al-2Ca	3.54±2.62	0.87±0.38
5vol% SiCp/Mg-5Al-2Ca	1.80±1.11	0.68±0.22
10vol% SiCp/Mg-5Al-2Ca	1.38±0.28	0.66±0.19

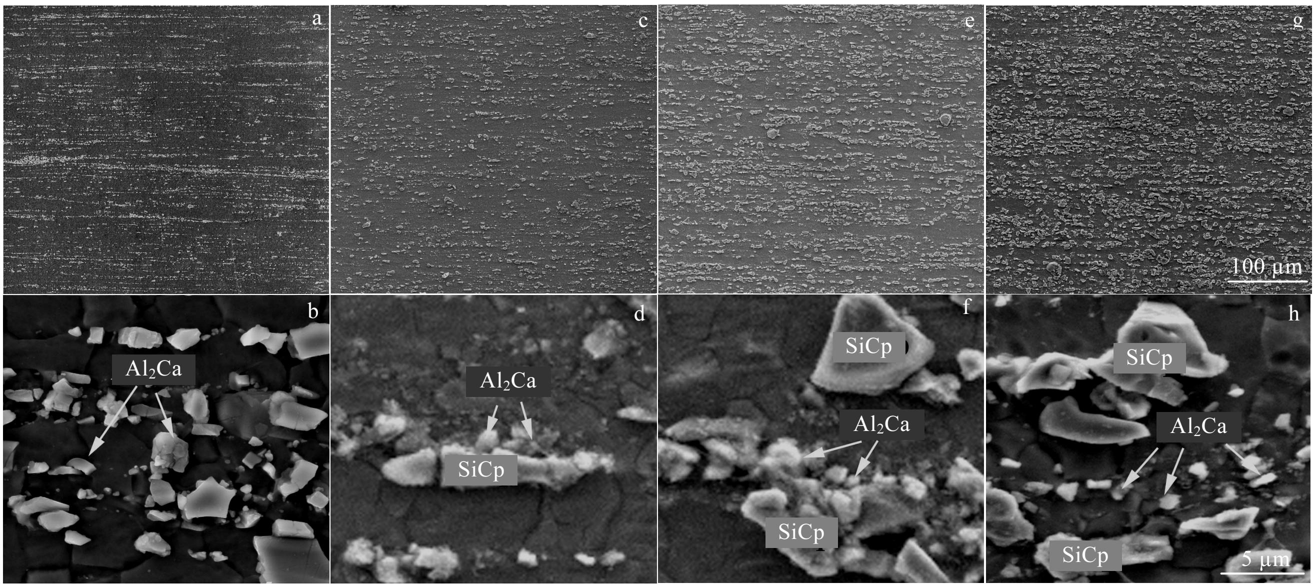

Fig.4 SEM images of as-extruded Mg-5Al-2Ca alloy (a, b) and composites with 2vol% (c, d), 5vol% (e, f) and 10vol% (g, h) SiCp

~3 μm, indicated by Fig.5b. As 2vol% SiCp is added, rare elongated grains can be observed in the composite and the matrix is mainly composed of fine equalized grains, as shown in Fig.5c. Fig.5d indicates that the average grain size of 2vol% SiCp/Mg-5Al-2Ca composite is ~2 μm. The smaller grain size of the SiCp/Mg-5Al-2Ca composite implies that addition of a small amount of SiCp has obvious effect on grain refinement. Moreover, the average grain size decreases slightly as the SiCp content increases from 2vol% to 10vol%, as illustrated in Fig. 5e~5h.

2.3 Mechanical properties

The tensile stress-strain curves of as-extruded Mg-5Al-2Ca alloy and SiCp/Mg-5Al-2Ca composites are depicted in Fig.6a. The tensile yield strength (TYS), ultimate tensile strength (UTS) and elongation obtained from Fig.6a are summarized in Fig.6b. It is obvious that small addition of SiCp could improve effectively the mechanical properties of composites. The TYS increases as the volume fraction of SiCp increased from 2% to 10% while the elongation decreases. Moreover, the UTS of composites does not change monotonously with increasing of SiCp content. The highest value of UTS appears when the volume fraction of SiCp is 5%.

3 Discussion

3.1 SiCp's effect on Al_2Ca phase

The Al_2Ca phase which formed during solidification process prefers to precipitate along grain boundaries and results in the network distribution after solidification[17]. Owing to the existence of surface energy between SiCp and Mg melt in the composite, the solute's free energy of liquid in contact with particle surface is higher than that in the melt[18]. In order to keep the thermodynamic equilibrium, the Al and Ca atoms tend to concentrate at the interface between SiCp and Mg. Thus, the Al_2Ca phase prefers to precipitate at the surface of SiCp as compared with grain boundaries and the network distribution of Al_2Ca phase is destroyed. Consequently, the gradual refinement of Al_2Ca phase in composites can be attributed to the more nucleation sites introduced by the increasing of reinforcement volume fraction.

According to Lloyd's investigation[19], the stress concentration occurs easily near larger particles as compared with fine ones in metal matrix composite. Therefore, for the present investigation, the SiCp with a larger size will bear greater stress as compared with Al_2Ca phase and tend to break during extrusion. Moreover, the Al_2Ca phase in the composite containing more SiCp is finer than that of the composite with a small amount of SiCp addition. Thus, reduction of the Al_2Ca size after extrusion in 10vol% SiCp/Mg-5Al-2Ca composite is not as obvious as that of the composite with 2 vol% SiCp, as mentioned in section 2.2.

3.2 Microstructure influenced by SiCp and Al_2Ca phase

Normally, the DRX caused by deformation in particle reinforced magnesium matrix composite is strongly influenced by the particle size. Two main effects are introduced by the reinforcement particles on grain refinement: i) the formation of particle deformation zone around micron particles [20] and ii) the pinning effect caused

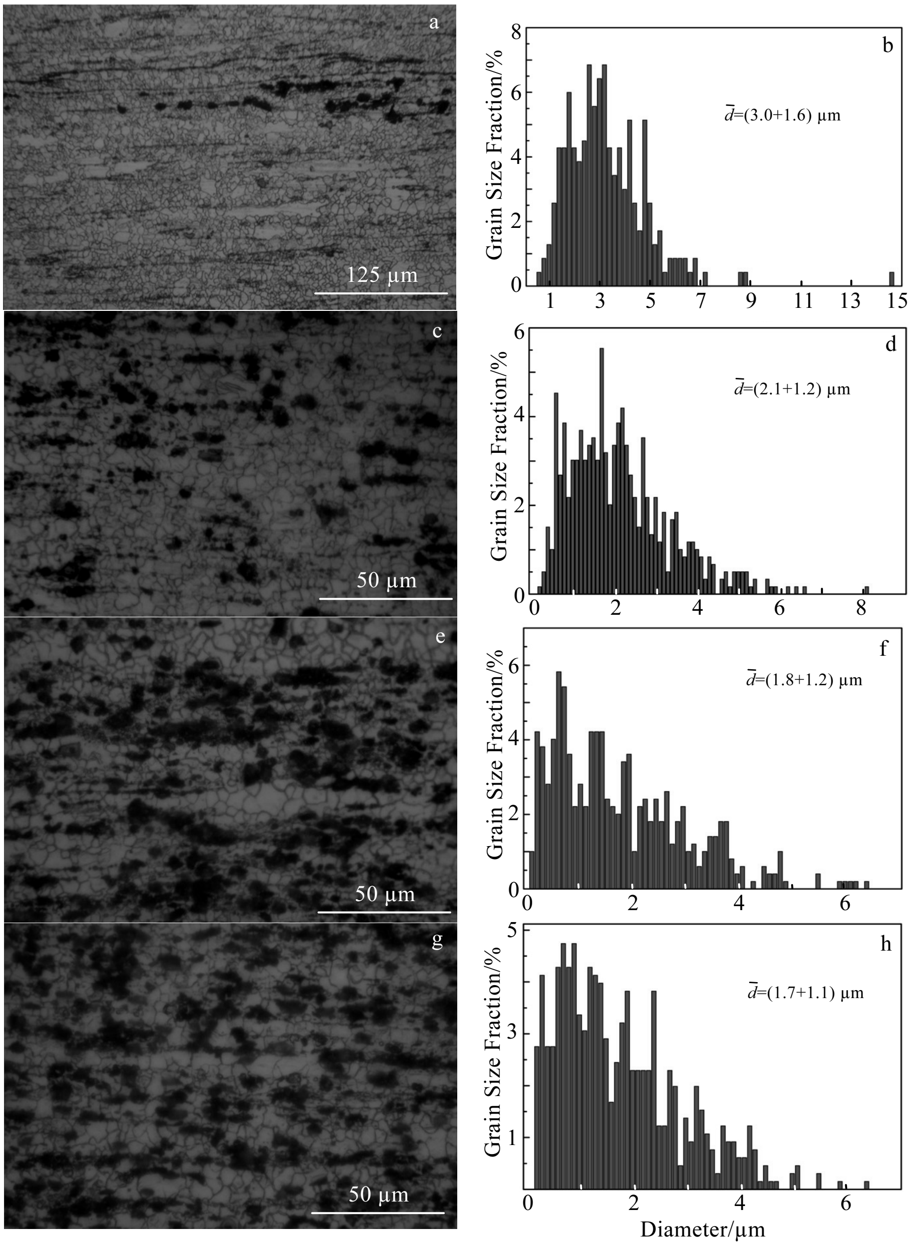

Fig.5 Optical microstructures (a, c, e, g) and grain size distribution (b, d, f, h) of as-extruded Mg-5Al-2Ca alloy (a, b) and SiCp/Mg-5Al-2Ca composites with 2vol% (c, d), 5vol% (e, f) and 10vol% (g, h) SiCp

by submicron particles on grain growth. In bimodal reinforcement system (submicron + micron) SiCp/AZ91 composite, grains could be refined by stimulated DRXed nucleation caused by micron particles and the hindered grain boundary movement results from submicron particles during hot extrusion, while the micron particles play a dominant role for grain refinement [21]. In the present study, it can be assumed that addition of micron SiCp is the main reason for grain refinement. Moreover, the fine Al_2Ca phase, which is supposed having the pinning effect on grain boundary migration, is distributed uniformly in the composites. Thus, the grain refinement is more obvious in the composites in the present study.

It is interesting that decreasing grain size of the composite would be expected with increasing of SiCp volume fraction in composite whereas no obvious changing of grain size can be observed in the case, as shown in Fig. 5. Generally, grain size (d) of the DRXed grains during extrusion can be described as $d=C(G/N)^{1/4}$, where C is a constant, N is DRX nucleation rate and G is growth rate. As the volume fraction of SiCp increases from 2% to 10%, despite of the high N value introduced by the increasing amount of SiCp, G would also increase due to more heat generated during extrusion by addition of a large number of secondary phases[22]. Consequently, the grain size of composite does not decrease obviously when the SiCp increases from 2vol% to 10vol%.

3.3 Mechanical properties influenced by SiCp and Al_2Ca phase

According to Hall–Petch relation [13], the yield strength increases with decreasing of grain size. Moreover, due to the different coefficients of thermal expansion (CTE) between particle and matrix, high density dislocation occurs around particles during the cooling process of extrusion. Therefore, the increased TYS ($\Delta\sigma_{CTE}$) caused by the additional dislocation strengthening effect can be introduced by the mismatch of CTE[23,24]. Consequently, the improved TYS can be achieved in the composites as compared with Mg-5Al-2Ca alloy due to the addition of SiCp and decreased grain size. According to above section, the variation of grain size is not obvious in SiCp/Mg-5Al-2Ca composites as the volume fraction of SiCp increases from 2% to 10%. Thus, the increasing amount of volume fraction for SiCp can be considered as the dominant reason for the increased TYS of SiCp/Mg-5Al-2Ca composite.

After yielding, the tensile process is accompanied by the multiplication of dislocations which is the characteristic of work hardening [24]. Work hardening behavior of a material can be described by means of macroscopic work hardening rate: θ=dσ/dε, where σ and ε is the macroscopic true stress and true strain, respectively[25,26]. Based on the tensile stress-strain curves in Fig.6a, the work hardening rate of as-extruded Mg-5Al-2Ca alloy and SiCp/Mg-5Al-2Ca composites is plotted in Fig.7. It is obvious that the θ value of as-extruded SiCp/Mg-5Al-2Ca composites strongly depends on volume fraction of SiCp. As mentioned above, high dislocation density can be introduced by the increasing SiCp content owing to the different CTE values between particles and Mg matrix[23]. Moreover, the dislocation movement will be hindered by particles as well. Thus, the improved UTS of the composite can be attributed to the high working hardening rate caused by the high dislocation intensity which is related to the flow stress in present investigation.

SEM microstructures of fracture and longitudinal section near fracture surface for the as-extruded Mg-5Al-2Ca alloy and SiCp/Mg-5Al-2Ca composites are summarized in Fig.8. In the Mg-5Al-2Ca alloy, the cracked Al_2Ca phases along extrusion direction are observed near fracture surface, as indicated by the red arrows in Fig.8a. It can be concluded that the fracture happens during tensile process owing to the intrinsic brittleness of Al_2Ca phases. The ductile fracture features with numerous dimples and tear ridges can be observed on the fracture surface of monolithic Mg-5Al-2Ca alloy, as shown in Fig.8b. For the composites, although the addition of micron SiCp is propitious to the refinement and redistribution of Al_2Ca phase, the interfacial de-bonding between matrix and SiCp or Al_2Ca phase and cracked

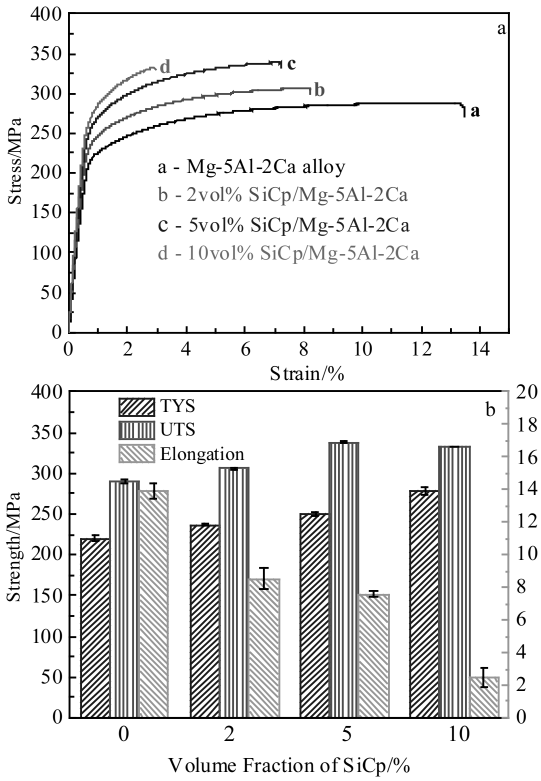

Fig.6 Mechanical properties of as-extruded Mg-5Al-2Ca alloy and SiCp/Mg-5Al-2Ca composites: (a) tensile stress-strain curves; (b) TYS, UTS and elongation

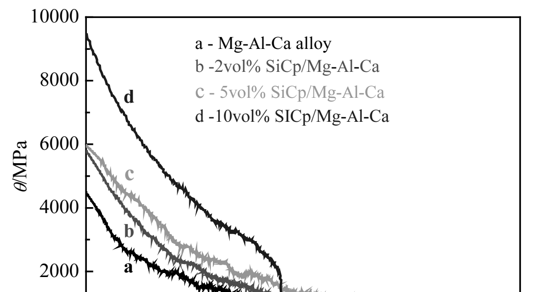

Fig.7 Work hardening rate (θ) as a function of net flow stress ($\sigma - \sigma_{0.2}$) of as-extruded Mg-5Al-2Ca alloy and SiCp/Mg-5Al-2Ca composites

particles may serve as a crack source and lead to fracture of the composite during tensile process, as shown in Fig.8c, 8e and 8g. Fig.8d, 8f and 8h reveal the fracture surfaces of composites with different volume fractions of SiCp, indicating the typical brittle fracture is surrounded by the ductile dimples of the surrounding matrix. Generally, the existence of a large amount of particles can lead to large

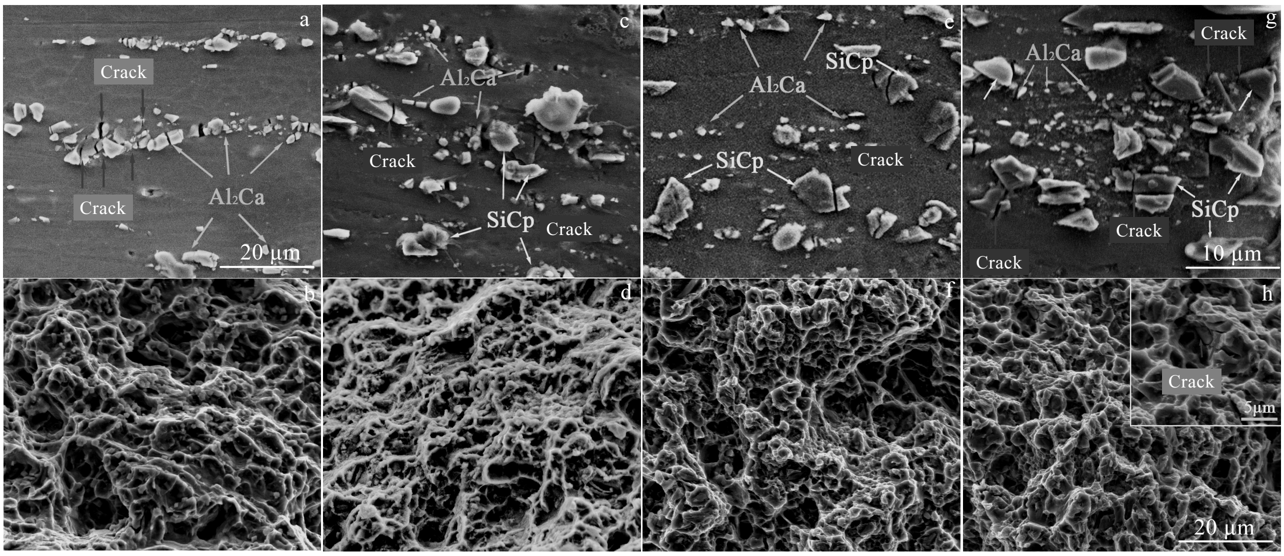

Fig.8 SEM microstructures of longitudinal section near fracture surface (a, c, e, g) and fractography (b, d, f, h) for the as-extruded alloy (a, b) and composites with 2vol% (c, d), 5vol% (e, f), and 10vol% (g, h) SiCp

stress concentration due to high dislocation density. Therefore the microcracks appear readily in the composite with a large amount of particles during tensile process. Thus, elongation of the composites is reduced with increasing of the SiCp volume fraction. Moreover, it could be deduced that the minor drop of UTS in 10vol% SiCp/Mg-5Al-2Ca composite as compared with the composite with 5vol% SiCp composite may also result from the quick propagation of microcracks caused by the large stress concentration.

4 Conclusions

1) Addition of SiCp is propitious to disturb the network distribution of Al_2Ca phases in the as-cast alloy. The network distribution disappears and Al_2Ca phase is refined gradually with increasing of SiCp content.

2) Grains and Al_2Ca phase are refined obviously in both the monolithic Mg-5Al-2Ca and SiCp/Mg-5Al-2Ca composites after hot extrusion. The variation of Al_2Ca phase and grain size is unobvious as the SiCp content increases from 2% to 10%.

3) Addition of SiCp leads to obvious improvement of TYS, UTS and θ value, however, at the expense of elongation. The UTS of SiCp/Mg-5Al-2Ca composite does not change monotonously with increasing SiCp content, and the highest UTS appears at the volume fraction of 5%.

4) The interfacial de-bonding between matrix and SiCp or Al_2Ca phase and cracked particles may serve as a crack source and lead to fracture of the composite during tensile process.

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SiCp 颗粒含量对 SiCp/Mg-5Al-2Ca 复合材料组织与性能的影响

常　海[1]，王翠菊[2]，邓坤坤[2]，聂凯波[2]，苏　坤[2]，廖　玲[2]，王红霞[2]，佟立波[3]

(1. 北京科技大学 国家材料服役安全科学中心，北京 100083)

(2. 太原理工大学 先进镁基材料山西省重点实验室，山西 太原 030024)

(3. 中国科学院长春应用化学研究所 稀土资源利用国家重点实验室，吉林 长春 130022)

摘　要：通过搅拌铸造法制备了 3 种不同体积分数（2%，5%，10%）的 SiCp/Mg-5Al-2Ca 复合材料，并在 673 K 下进行了热挤压。铸态复合材料中，少量 SiCp 颗粒的加入就能破坏 Al_2Ca 相沿基体合金晶界分布并有效细化 Al_2Ca 相析出尺寸。随着 SiCp 体积分数的增高，Al_2Ca 相尺寸有所减小，但不明显。经过热挤压后，Al_2Ca 相破碎并沿挤压方向排布，基体合金晶粒得到细化。晶粒尺寸以及 Al_2Ca 相尺寸随着 SiCp 体积分数的增高呈微小减小。与单组元基体合金相比较，挤压态 SiCp/Mg-5Al-2Ca 复合材料的屈服强度和加工硬化率随着 SiCp 体积分数的增高而逐渐增高，而延伸率则逐渐下降；抗拉强度最大值则出现在 SiCp 体积分数为 5%时。复合材料中 SiCp 颗粒以及 Al_2Ca 相的脱粘以及开裂是导致复合材料断裂的主要原因。

关键词：镁基复合材料；SiCp；增强相；显微组织；机械性能

作者简介：常　海，男，1983 年生，博士，助理研究员，北京科技大学材料服役安全科学中心，北京 100083，电话：010-62333860，E-mail: hchang@ustb.edu.cn