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# Determination of $\alpha/\beta$ phase boundaries and mechanical characterization of Mg-Sc binary alloys



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#### ABSTRACT

In this study,  $\alpha(hcp)/\beta(bcc)$  phase boundaries of the Mg-Sc binary phase system were determined by Mg/Sc diffusion couple and conventional equilibrated alloy methods. It was confirmed that  $\alpha/\beta$  phase boundaries exist in regions with a higher Mg content than those in reported diagram. The Mg-Sc alloy with a  $\beta$  single-phase showed tensile strength of 254 MPa and elongation of 25.4%. The tensile strength of the Mg-Sc alloy increased with increasing volume fraction of the  $\alpha$  phase ( $f_{\alpha}$ ) and the alloy with a  $f_{\alpha}$  of 34% showed high UTS of 310 MPa and elongation of 28.8%, properties which are better balanced than those of conventional Mg alloys. This improvement was suggested to be due to isotropic deformation of the  $\beta$  phase, small grain size and small c/a of the  $\alpha$  phase.

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#### 1. Introduction

Magnesium alloys, which are characterized by have light weight and high specific strength, are attracting attention as structural materials for electronics, automotive and aviation applications. Mg-Al-Zn alloys, such as AZ31 and AZ91 have been widely commercialized for electronics and automotive applications because of their good castability [1,2]. It is known that the addition of rare earth elements (RE) to Mg alloy is very effective to enhance its strength and a high yield stress of about ~200 MPa has been obtained in Mg-RE-Y systems [2]. Moreover, Mg-TM-RE alloys (TM: Ni, Cu, Zn; RE: Y, Gd, Dy, Ho, Er, Tb, Tm) which have a long-period stacking ordered (LPSO) phase have been also reported to show a high yield stress of over 300 MPa [3,4]. However, the ultimate tensile elongation of most Mg alloys is poor at room temperature due to the limited slip systems of the hexagonal close packed (hcp) structure [2,3]. It is well known that Mg cannot deform isotropically because of its anisotropic structure and, therefore, deformation twinning occurs to relax the anisotropic slip deformation. Meanwhile, it has been reported that the formation of  $\{10\overline{1}1\}$ - $\{10\overline{1}2\}$  double twins leads to premature failure because localized deformation in the double twins causes large surface steps and cracks [5]. In contrast, Mg binary alloys with a small amount of RE, such as Mg-Ce and Mg-Y alloys, show a much higher elongation but its strength is as low as that of pure Mg [6-8]. These facts suggest that Mg alloys with an hcp structure do not exhibit

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both high strength and high ductility. Therefore, development of Mg alloys with an isotropic crystal structure is desirable.

Among all Mg-X binary systems, only Mg-Li and Mg-Sc alloys have a  $\beta$  phase with a body centered cubic (bcc) structure in a Mgrich composition. Alloys with a bcc structure (β phase, Im-3m (229)) can be deformed more isotropically than those with an hcp structure ( $\alpha$  phase,  $P6_3/mmc$  (194)) because a bcc structure has more slip systems than an hcp structure. Actually, it has been reported that Mg-Li alloys with a  $\beta$  phase show an excellent ductility [9,10]. Meanwhile, there are a few reports on mechanical properties of Sc-added Mg alloys with single  $\alpha$  phase [11–13]. Very recently, the present authors have found that Mg-Sc alloy with a  $\beta$ phase shows significant age hardening due to the formation of very fine  $\alpha$  precipitates in  $\beta$  matrix [14]. Moreover, it has been demonstrated that age-hardened Mg-Sc alloy with an  $\alpha+\beta$  twophase microstructure shows high tensile strength of over 300 MPa and better ultimate tensile elongation of over 10% [15]. However, there has still not been any systematic study about the effect of the volume fraction of  $\alpha$  and  $\beta$  phases on the mechanical properties in Mg-Sc alloys.

There are some reported phase diagrams of the Mg-Sc binary system, but they differ from each other and contain unclear parts [16–18]. For example, phase boundaries of  $\alpha/\beta$  are different between phase diagrams reported by Beaudry and Daane in 1969 [16] and those reported by Nayeb-Hashemi and Clark in 1986 [17].

Given this background, the phase boundaries of  $\alpha/\beta$  in Mg-Sc binary phase diagram were reconsidered in this study. Furthermore, based on the obtained phase diagram, Mg-Sc alloys with various microstructures, such as  $\beta$  single-phase,  $\alpha+\beta$  two-phase

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and  $\alpha$  single-phase, were prepared by changing heat treatment conditions and tensile testing was carried out to investigate their mechanical properties.

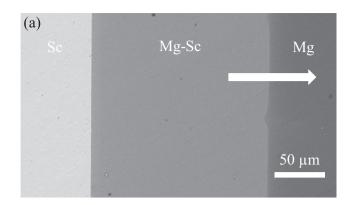
#### 2. Materials and methods

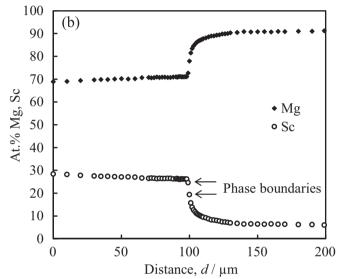
We need to know the exact position of  $\alpha/\beta$  phase boundaries to control the microstructure of Mg-Sc alloys. Thus, we investigated the  $\alpha/\beta$  phase boundaries of the Mg-Sc binary system using the diffusion couple method and the conventional equilibrated alloy method. Annealing conditions are summarized in Table 1. Diffusion couples were prepared as follows: Bulk pieces of Mg and Sc (99.99%) were mechanically polished in order to remove the oxidation layer. The polished Mg and Sc were enclosed in a quartz tube in an Ar atmosphere, and were subsequently annealed at 973 K to melt only the Mg piece. This sample was then cut into small pieces to obtain diffusion couples. The obtained diffusion couples were annealed at 773, 823 and 873 K for 96, 72 and 72 h, respectively, in an Ar atmosphere. For the conventional equilibrated alloy method, Mg-Sc alloys having a nominal composition of 15, 20 and 21 at% Sc were prepared by induction melting under Ar atmosphere. The ingot was cut into small pieces and then heat treated at 773, 823, 873, 953 and 963 K for certain times followed by water quenching. The compositions were measured by an electron probe microanalyzer (EPMA) with accelerating voltage of 15 kV. Microstructures of the samples were observed by optical microscope (OM) and scanning electron microscope (SEM). The crystal structure was analyzed by X-ray diffraction (XRD) operated at 40 kV and 40 mA using a Cu-K $\alpha$  source. The volume fraction of the  $\alpha$  phase ( $f_{\alpha}$ ) was evaluated from backscattered electron images using graphics software "image]". Grain size was determined by the intercept method from the OM images or SEM images of the sample surface after chemical etching [19], where the composition of etchant was nitric acid:acetic acid:distilled water:ethanol=1:3:4:12 [20].

Based on the phase diagrams obtained in this study, we investigated the mechanical properties of the Mg-20 at% Sc alloy with various volume fractions of the  $\alpha$  and  $\beta$  phases, which can be obtained by changing the heat treatment temperature. The Mg-20 at% Sc alloy ingot melted in a high frequency induction furnace was hot rolled at 873 K and then cold rolled until a thickness of about 0.7 mm with annealing at 873 K. The samples for tensile testing were cut out by a wire electric discharge machine, where the gauge length and width being 10 and 3.6 mm, respectively. The samples were annealed at 773, 823, 873, 898 and 963 K for 10 min. Tensile testing was carried out using Shimazu AUTOGRAPH AG-1 at a strain rate of  $10^{-3}~\text{s}^{-1}$ . The tensile loading direction was parallel to the rolling direction. For comparison, tensile testing of AZ31 was also carried out.

Equilibrated Sc compositions at various temperatures obtained from diffusion couple and conventional equilibrated alloy methods.

		Temperature (K)	Time	Composition of Sc (at%)	
				α	β
Diffusion couple		773 823 873	96 h 72 h 72 h	19.5 18.6 16.6	24.7 22.8 21.6
Equilibrated alloy method	Two- phase Single- phase	773 823 873 953 963	96 h 72 h 24 h 24 h 30 min	20.5 18.4 16.3 13.5	26.5 24.5 22.0 17.6 19.4





**Fig. 1.** (a) Cross-sectional SEM image and (b) composition profile of a Mg/Sc diffusion couple treated at 773 K for 96 h.

#### 3. Results and discussion

#### 3.1. Determination of $\alpha/\beta$ boundaries

Fig. 1(a) shows the backscattered electron image of the Mg/Sc diffusion couple annealed at 773 K for 96 h. An intermediate layer was formed between Mg and Sc. In other diffusion couples, only an intermediate layer was also observed between Mg and Sc. The composition profile of the diffusion couple measured by EPMA along the white line indicated in Fig. 1(a) is shown in Fig. 1(b). The Mg/Sc composition ratio of the intermediate layer was about 7/3, indicating the  $\beta$  phase. As shown in Fig. 1(b), clear jumps of concentration of Mg and Sc are observed at Mg/Mg-Sc interface. The compositions at clear jumps indicated by arrows in Fig. 1(b) are considered to be the phase boundaries. We measured the composition profiles at 5 lines and averaged the obtained values. The obtained results are listed in Table 1.

Constituent phases of equilibrated samples at several conditions, as shown in Table 1, were confirmed by means of XRD and SEM. Fig. 2(a) shows the XRD pattern of the sample annealed at 873 K for 24 h. At 873 K, the sample has  $\alpha+\beta$  dual-phase structure. A backscattered electron image of this sample is also shown in Fig. 2(b). The dark-colored region denotes the  $\alpha$  phase and the bright-colored region denotes the  $\beta$  phase. The equilibrium concentrations measured by EPMA of all samples are summarized in Table 1.

The results obtained by diffusion couple and conventional equilibrated alloy methods are plotted on the Mg-Sc phase

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