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Experimental investigation of phase equilibria in the Mg-rich corner of Mg-Nd-Sc system

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Abstract

Phase equilibria in the Mg-rich corner of Mg–Nd–Sc ternary system was studied by using the equilibrated alloys method. The partial isothermal sections at 500 °C, 530 °C, and 550 °C were constructed by analyzing the phase constitution and the chemical compositions of the present phases in 19 alloy samples. The Mg3(Mg, Nd, Sc) phase exhibited noticeable ternary solubility introduced by the solid solution of Mg and Sc in the Mg3Nd lattice. (Mg_hcp) showed a narrow homogeneity range paralleling to Mg–Sc binary boundary, whereas Mg41Nd5 showed negligible ternary solubility at all three temperatures. The three-phase equilibrium region of (Mg_hcp) + Mg41Nd5 + Mg3(Mg, Nd, Sc) dominated the Mg-rich corner and was surrounded by three two-phase equilibrium regions constituted by any two of above three phases. The primary crystals and the solidification pathways of alloys in the Mg-rich corner were analyzed via the as-cast microstructure. The matrix phases of (MgSc_bcc) and/or (Mg_hcp) together with the precipitations of Mg3(Mg, Nd, Sc) and other ordering structures could provide additional space to optimize microstructure and properties of Sc-alloyed Mg–Nd alloys.

1. Introduction

Rare Earth (RE) Mg alloys have much more attention due to high strength, improved creep resistance, and good deformability [1–3]. Nd and other RE elements are commonly utilized due to the pronounced precipitation strengthening [6–8] and the modest solid solution strengthening [9, 10]. The most successful Mg alloys developed in this category are Mg–Nd–Y system [7, 11, 12] and the modified versions by adding Tb, Er, Dy, Gd, etc. [1–5]. Sc element exhibits physical and chemical properties similar to RE elements but a lower density. The solubility of Sc in Mg lattice is wider than common RE elements. Alloying with Sc increases the melting temperature [13] and decreases the diffusivity of Mg-based solid solution [(Mg_hcp) hereinafter] [14], which would be in favor of solid solution strengthening and creep resistance [15]. The formation of ordered precipitates involving Mg and Sc may also improve the room- and high-temperature properties [16–19]. Therefore, the design of the ternary Mg–Nd–Sc even multi-component Mg alloys by adjusting the ratio of Sc to Nd is expected to enhance the effect of age-hardening in RE contained Mg alloys.

Phase diagram has served as a roadmap for optimizing composition, microstructure, and processing [20, 21]. It conveys useful information on the solid solubility limit and the precipitated phase being equilibrium with the matrix. The binary phase diagrams of Mg–Nd [22], Mg–Sc [13, 23], and Nd–Sc [24] have been experimentally studied [25–27] and assessed via the technique of computational thermodynamics [13, 23, 28–31]. Different from Mg–Nd–Y system [7, 32], the phase diagram data of the Mg–Nd–Sc ternary system are still limited. The objective of the present work is to experimentally determine the phase equilibrium relations in the Mg–Nd–Sc ternary system and construct the equilibrium isothermal sections at 500 °C, 530 °C, and 550 °C.
by studying the equilibrium phase constitutions and the chemical compositions of equilibrium phases in alloys. The determined ternary phase boundaries, solid solubilities, and solidification pathways will provide the fundamental data to guide the composition optimization and phase selection in the works of solid solution strengthening and/or precipitation strengthening.

2. Experiments

19 ternary alloy samples were prepared to study the phase equilibria in Mg–Nd–Sc system and to construct the isothermal sections at 500 °C, 530 °C, and 550 °C. The alloys were prepared from the binary master alloys of Mg-29.85 wt.% Sc and Mg-23.94 wt.% Nd together with pure Mg (99.99 wt.%) and pure Nd (99.9 wt.% Nd). Due to the high melting temperature of Nd, the pure Nd grains with a size smaller than 1 mm × 1 mm were utilized to facilitate the melting. The weighed raw materials were wrapped in inner tubes made of tantalum foils (twisted at both ends) and then sealed in outer tubes made of quartz under argon atmosphere. The two-layer sealed tubes were heated up and held at 950 °C for 4 h for melting in a muffle furnace. To promote chemical composition homogeneity, the tubes were turned upside down every 1 h. The ingots with good quality were resealed in the argon backfilled quartz capsules and annealed at 500 °C, 530 °C, and 550 °C for 720 h, 672 h, and 240 h, respectively. The alloy samples after annealing were quickly pulled out from the furnaces and quenched into ice water to retain the microstructure and composition distribution at the annealing temperatures.

Table 1. Chemical compositions of alloys, equilibrium phase identification, and chemical compositions of phases at 500 °C.

| No. | Mg  | Nd  | Sc  | SEM/EPMA phase ID      | Mg  | Nd  | Sc  | XRD phase ID      |
|-----|-----|-----|-----|------------------------|-----|-----|-----|------------------|
| 1   | 93.8| 4.1 | 2.1 | (Mg)                   | 96.8| 0.3 | 2.9 | (Mg)             |
|     |     |     |     | Mg3Nd                 | 88.8| 11.2| 0   | Mg3Nd            |
| 2   | 91.6| 2.4 | 6.0 | (Mg)                   | 92.1| 0.4 | 7.5 | (Mg)             |
|     |     |     |     | Mg3Nd                 | 88.8| 11.1| 0.1 | Mg3Nd            |
| 3   | 92.5| 6.9 | 0.6 | (Mg)                   | 98.9| 0.3 | 0.8 | (Mg)             |
|     |     |     |     | Mg3Nd                 | 88.8| 11.2| 0   | Mg3Nd            |
| 4   | 90.3| 8.2 | 1.5 | (Mg)                   | 94.0| 0.3 | 5.7 | (Mg)             |
|     |     |     |     | Mg3Nd                 | 88.5| 11.3| 0.2 | Mg3Nd            |
| 5   | 89.7| 3.0 | 7.3 | (Mg)                   | 89.5| 0.5 | 10.0| (Mg)             |
|     |     |     |     | Mg3Nd                 | 77.9| 20.2| 1.9 | Mg3Nd            |
| 6   | 89.2| 5.3 | 5.3 | (Mg)                   | 89.3| 0.4 | 10.3| (Mg)             |
|     |     |     |     | Mg3Nd                 | 77.9| 20.2| 1.9 | Mg3Nd            |
| 7   | 88.2| 8.6 | 3.2 | (Mg)                   | 88.9| 10.8| 0.3 | Mg3Nd            |
|     |     |     |     | Mg3Nd                 | 77.9| 20.2| 1.9 | Mg3Nd            |
| 8   | 87.0| 9.8 | 3.2 | (Mg)                   | 88.9| 0.3 | 10.8| (Mg)             |
|     |     |     |     | Mg3Nd                 | 88.2| 11.5| 0.3 | Mg3Nd            |
| 9   | 85.3| 11.8| 2.9 | (Mg)                   | 89.2| 0.4 | 10.4| (Mg)             |
|     |     |     |     | Mg3Nd                 | 77.5| 20.3| 2.2 | Mg3Nd            |
| 10  | 82.8| 2.5 | 14.7| (Mg)                   | 83.5| 0.4 | 16.1| (Mg)             |
|     |     |     |     | Mg3Nd                 | 76.4| 20.5| 3.1 | Mg3Nd            |
| 11  | 82.2| 5.3 | 12.5| (Mg)                   | 83.8| 0.4 | 15.8| (Mg)             |
|     |     |     |     | Mg3Nd                 | 76.5| 20.5| 3.0 | Mg3Nd            |
| 12  | 80.7| 11.5| 7.8 | (Mg)                   | 84.9| 0.4 | 14.7| (Mg)             |
|     |     |     |     | Mg3Nd                 | 77.1| 20.2| 2.7 | Mg3Nd            |
| 13  | 79.5| 4.8 | 15.7| (Mg)                   | 80.2| 0.3 | 19.5| (Mg)             |
|     |     |     |     | Mg3Nd                 | 75.8| 20.6| 3.6 | Mg3Nd            |
|     |     |     |     | Mg3Sc                 | 49.3| 0   | 50.7| w/o              |
The phase present in the annealed alloys were examined by powder X-ray diffraction (XRD) using a Rigaku Smartlab X-ray diffractometer and Scanning Electron Microscopy (SEM) using JEOL JSM-6510. XRD was performed with Cu Kα radiation at 40 kV, 200 mA, in the range of 2θ from 10° to 80°, and a rate of 4° min⁻¹. The chemical composition of the alloys and that of the individual phases were detected using Energy Dispersive x-ray Spectroscopy (EDS) and Electro-Probe Microanalyzer (EPMA) equipped on JEOL JXA-8230, respectively. The acceleration voltage was set 15 kV and the beam current was 20 × 10⁻⁸ A. The chemical composition was detected in different areas of a sample repetitively, thus the mean values were of statistical significance.

Tables 1–3 summarize the actual chemical compositions of the alloys and the identifications of individual phases as well as the chemical composition of each phase.

### Table 2. Chemical compositions of alloys, equilibrium phase identification, and chemical compositions of phases at 530 °C.

| No. | Mg (at.%) | Nd (at.%) | Sc (at.%) | SEM/EPMA phase ID | Chemical composition of phases (at.%) | XRD phase ID |
|-----|-----------|-----------|-----------|-------------------|--------------------------------------|--------------|
|     | Mg        | Nd        | Sc        |                   | MgNd/Mg41Nd5                          | Mg41Nd5      |
| 14  | 96.5      | 2.4       | 1.1       | Mg41Nd5           | 97.8                                 | (Mg)         |
|     |           |           |           |                   | 88.8                                 | Mg41Nd5      |
| 1   | 93.8      | 4.1       | 2.1       | Mg41Nd5           | 96.1                                 | (Mg)         |
|     |           |           |           |                   | 88.5                                 | Mg41Nd5      |
| 6   | 89.2      | 5.5       | 5.3       | Mg41Nd5           | 91.3                                 | (Mg)         |
|     |           |           |           |                   | 88.6                                 | Mg41Nd5      |
| 15  | 89.5      | 2.4       | 8.1       | Mg3Nd             | 79.7                                 | Mg3Nd        |
|     |           |           |           |                   | 90.7                                 | Mg3Nd        |
| 16  | 83.8      | 3.5       | 12.7      | Mg3Nd             | 79.4                                 | Mg3Nd        |
|     |           |           |           |                   | 90.7                                 | Mg3Nd        |
| 12  | 80.7      | 11.5      | 7.8       | Mg3Nd             | 77.1                                 | Mg3Nd        |
|     |           |           |           |                   | 77.7                                 | Mg3Nd        |
| 17  | 80.6      | 5.4       | 14.0      | Mg3Nd             | 77.1                                 | Mg3Nd        |
|     |           |           |           |                   | 59.3                                 | Mg3Nd        |

### Table 3. Chemical compositions of alloys, equilibrium phase identification, and chemical compositions of phases at 550 °C.

| No. | Mg (at.%) | Nd (at.%) | Sc (at.%) | SEM/EPMA phase ID | Chemical composition of phases (at.%) | XRD phase ID |
|-----|-----------|-----------|-----------|-------------------|--------------------------------------|--------------|
|     | Mg        | Nd        | Sc        |                   | MgNd/Mg41Nd5                          | Mg41Nd5      |
| 14  | 96.5      | 2.4       | 1.1       | Mg41Nd5           | 97.8                                 | (Mg)         |
|     |           |           |           |                   | 88.7                                 | Mg41Nd5      |
| 1   | 93.8      | 4.1       | 2.1       | Mg41Nd5           | 95.9                                 | (Mg)         |
|     |           |           |           |                   | 88.8                                 | Mg41Nd5      |
| 2   | 91.6      | 2.4       | 6.0       | Mg41Nd5           | 91.8                                 | (Mg)         |
|     |           |           |           |                   | 88.5                                 | Mg41Nd5      |
| 4   | 90.3      | 8.2       | 1.5       | Mg41Nd5           | 94.7                                 | (Mg)         |
|     |           |           |           |                   | 88.5                                 | Mg41Nd5      |
| 6   | 89.2      | 5.5       | 5.3       | Mg41Nd5           | 91.4                                 | (Mg)         |
|     |           |           |           |                   | 80.5                                 | Mg41Nd5      |
| 18  | 84.4      | 2.7       | 12.9      | Mg41Nd5           | 85.3                                 | (Mg)         |
|     |           |           |           |                   | 78.2                                 | Mg41Nd5      |
| 10  | 82.8      | 2.3       | 14.7      | Mg41Nd5           | 83.5                                 | (Mg)         |
|     |           |           |           |                   | 77.6                                 | Mg41Nd5      |
| 19  | 80.6      | 8.9       | 10.5      | Mg41Nd5           | 83.0                                 | (Mg)         |
|     |           |           |           |                   | 77.4                                 | Mg41Nd5      |
3. Results and discussions

3.1. Isothermal section at 500 °C

Figure 1 shows the XRD patterns and back-scattered SEM images of representative alloy samples after annealing at 500 °C. The subplots (a) and (b) correspond to alloy 4#. The phase constitution was identified (Mg_hcp) + Mg41Nd5 in XRD patterns. The chemical composition of the grey block was detected Mg88.5Nd11.3Sc0.2 with a stoichiometric ratio close to Mg41Nd5. Thus, the remaining dark phase riched in Mg was inferred to be (Mg_hcp) phase. The subplots (c) and (d) demonstrate a three-phase equilibrium of (Mg_hcp) + Mg41Nd5 + Mg3Nd present in the alloy sample 8#. Although the long-term annealing at 500 °C failed to completely erase the history of peritectic solidification which was inherited from Mg–Nd binary, the argument of phase equilibrium was concluded from the polygonized shape of Mg41Nd5 phase and the repeatability of the chemical composition measurement for one same phase in different areas. The detected chemical composition of Mg3Nd showed a value of Mg77.9Nd20.2Sc1.9. The content of Sc indicated the occurrence of the ternary solid solution in the lattice of Mg3Nd. Similar chemical composition of Mg3Nd was also found in the alloy sample 12# which inferred a two-phase equilibrium of (Mg_hcp) + Mg3Nd (see figures 1(e) and (f)).

The isothermal section can be outlined by summarizing all the experimental data in table 1. Figure 2 portrays the equilibrium isothermal section of the Mg-rich Mg–Nd–Sc system at 500 °C. The chemical compositions of the samples and those of the phases present in them are marked in circles and crosses, respectively. The single-phase region of structure homogeneity is highlighted in yellow. (Mg_hcp) phase shows a narrow structure homogeneity range paralleling to Mg–Sc binary boundary. The saturated solid solubility of Nd in (Mg_hcp) is assessed around 0.5 at.%. The binary intermetallic compound Mg3Nd extends the region of structure homogeneity into the interior of the isothermal section by introducing the solid solution of Mg and Sc. The
molecular formula thus is expressed as Mg₃(Mg, Nd, Sc). The Mg₄₁Nd₅ phase shows negligible ternary solubility in Mg–Nd–Sc ternary, which is different from other Mg-RE-RE(RE-like) system [33–35]. There is only one three-phase equilibrium region of (Mg_hcp) + Mg₄₁Nd₅ + Mg₃(Mg, Nd, Sc) present in the Mg-rich corner. It is surrounded by three two-phase equilibrium regions constituted by any two of above three phases. These two-phase equilibria are illustrated by the green tie-lines. The chemical compositions of (Mg_hcp) phase in 1# ∼ 4# are varying with the chemical compositions of alloys, while that of Mg₄₁Nd₅ is almost fixed. The XRD results have demonstrated the same phase constitution of (Mg_hcp) + Mg₄₁Nd₅ in all these 4 samples. The reasoning also applies to alloy samples 10# ∼ 13# in the two-phase equilibrium of (Mg_hcp) + Mg₃(Mg, Nd, Sc). In figure 3, there are a small number of light grey blocks with the chemical composition of Mg₄⁹Nd₀₂Sc₅₀₈ in alloy 10# in addition to (Mg_hcp) and Mg₃(Mg, Nd, Sc). One may misjudge that the (MgSc_bcc) phase was

Figure 2. The partial isothermal section at 500 °C of the Mg–Nd–Sc system according to experimental data.

Figure 3. (a) SEM back-scattered electron image and (b) XRD pattern of the alloy 10# annealed at 500 °C.
equilibrium in alloy 10# and the alloy 10# was in a three-phase equilibrium region. However, the presence of (MgSc
bcc) phase can not be confirmed since the positions of the diffraction peaks of (MgSc
bcc) phase coincide with some diffraction peaks of (Mg_hcp) and Mg3Nd. The chemical compositions of (Mg_hcp) matrix are varying in samples 10#, 11#, and 13#. It deviates from the principle that the chemical composition of individual phases in the three-phase equilibrium triangle would be fixed, independent of alloy compositions. Therefore, we can reason that the alloys 10#, 11#, and 13# must be located in the two-phase equilibrium region of (Mg_hcp) + Mg3(Mg, Nd, Sc). The light grey blocks may be introduced by the diffusional transformation between the undissolved pure Sc (in hcp structure) and the Mg in (Mg_hcp).

3.2. Isothermal sections at 530 °C and 550 °C
The phase equilibria displayed on the isothermal sections of 530 °C and 550 °C are similar to 500 °C(see figures 4 and 5). A liquid phase is present at 550 °C in a narrow three-phase equilibrium region close to the Mg–Nd boundary binary. The (Mg_hcp), Mg41Nd5, and Mg3(Mg, Nd, Sc) remain present at both temperatures. The
region of (Mg_hcp) is shrinking along the Mg–Sc boundary with increasing temperature, whereas that of Mg₄₁(Mg, Nd, Sc) phase is subjected to slight expansion towards the Mg-rich corner. The chemical composition of (Mg_hcp) in the three-phase equilibrium triangle of (Mg_hcp) + Mg₄₁Nd₅ + Mg₃(Mg, Nd, Sc) is also left shifting towards the Mg-rich corner, from ∼23 at.% Sc at 500 °C to ∼18 at.% Sc at 550 °C.

Figure 6 shows the XRD patterns and SEM images of 14#, 6#, and 12# alloys after annealing at 530 °C. In comparison with figure 1, the increased temperature leads to growth and full crystallization of individual phases. In particular, the history of peritectic solidification for the 6# alloy in the three-phase equilibrium region has been completely erased.

Most of (Mg_hcp), Mg₄₁Nd₅, and Mg₃(Mg, Nd, Sc) grains show perfect crystallization of convex polygon shapes. The collision and coalesce of Mg₄₁Nd₅ grain boundaries are clearly shown. The morphology indicates a near-equilibrium state. Figure 7 illustrates SEM images and the XRD patterns of 14#, 1#, 6#, and 19# alloys after annealing at 550 °C. 1# and 14# alloys have the same phase constitution in the XRD patterns but display different morphologies in SEM observation. The morphology of 14# alloy exhibits the characteristics of as-cast microstructure after long-term annealing, indicating a full re-melting and re-solidification in this alloy. Therefore, alloys 14# is in equilibrium with the liquid at 550 °C. In contrast, the coarse grains of (Mg_hcp) and Mg₄₁Nd₅ are clearly shown in alloy 1#, indicating an equilibrium of (Mg_hcp) + Mg₄₁Nd₅. Different from the polygon-shaped phase boundary in the Mg-rich alloys, the phase boundaries of (Mg_hcp) and Mg₃(Mg, Nd, Sc) in 12# alloy at 530 °C and 19# alloy at 550 °C show irregular shape. These two alloys with chemical compositions relatively distant from the Mg corner are difficult to fully attain phase equilibrium due to the relatively high thermodynamic stability and the low diffusion coefficients of element in Mg₃(Mg, Nd, Sc) phase.
3.3. Primary crystallization and solidification pathways

In addition to the study of isothermal phase equilibria, a furnace cooling was employed to study the near-equilibrium solidification behaviors. The as-cast microstructure can convey rough but useful information on the primary crystallization and solidification pathways. Figure 8 summarizes the preliminary results of primary crystallization regions based on the observation/speculation in this work. Three fields of primary crystallization dominate the liquidus of the Mg-rich corner, i.e. (Mg\_hcp), Mg₃(Mg, Nd, Sc), and (Mg\_bccA2). Figure 9 shows the SEM images of the as-cast 1\# and 4\# alloys which depict different solidification pathways. The as-cast image of 1\# (figure 9(a)) illustrates the solidification pathway of the primary crystallization of (Mg\_hcp) followed by the eutectics of L → (Mg\_hcp) + Mg₄₁Nd₅. The microstructure of 4\# in figure 9(b) is consist of primary crystal of Mg₃(Mg, Nd, Sc), peritectic layer Mg₄₁Nd₅, and eutectics of (Mg\_hcp) + Mg₄₁Nd₅. It’s worth noting that the Mg₁₂Nd phase commonly found in as-cast Mg-Nd alloy hasn’t been detected in our experiment. The small cooling rate in the furnace may suppress the formation of Mg₁₂Nd.

Figure 7. (a), (c), (e), (g) SEM back-scattered electron images and (b), (d), (f), (h) XRD patterns of the alloys 14\# (a), (b), 1\# (c), (d), 6\# (e), (f), and 19\# (g), (h) after being annealed at 550 \(^{\circ}\)C for 10 days.

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In addition, an alloy Mg_{92.6}Nd_{7.2}Sc_{0.2} (denote X in figure 8) was prepared to verify the existence of Mg_{41}Nd_{5} primary crystallization. Figure 10 shows the as-cast microstructure and XRD pattern. The alloy has undergone the primary crystallization of Mg_{41}Nd_{5} followed by the eutectics of L\rightarrow (Mg\_hcp) + Mg_{41}Nd_{5} (figure 10(a)). Other than the conventional lamellar eutectics, the coarse blocks of the (Mg\_hcp) phase are observed in figure 10. The formation of the block (Mg\_hcp) phase is probably triggered by the divorced eutectic mechanism considering the much smaller size of Mg_{41}Nd_{5} primary crystallization region than (Mg\_hcp). The small cooling rate in the furnace can further facilitate the growth of (Mg\_hcp) blocks. The experimental discoveries indicate that the above three alloys follow the solidification pathway similar to Mg–Nd binary alloys since their chemical compositions are close to the Mg–Nd boundary binary. Figure 11 depicts a complex as-cast microstructure of 13\# which combines solidification reactions and solid states phase transformations. The coarse dendrites of the black phase with convex boundaries imply the nucleation and growth of them from the liquid phase. The chemical composition of the black phase is detected Mg_{83.5}Nd_{0.8}Sc_{15.7}, far away from the alloy composition. A large number of particles are dispersely precipitated in the black matrix. All these experimental phenomena indicate the occurrence of element partitioning and diffusional solid states phase transformations after primary crystallization. The white phase is identified as Mg_{x}(Mg, Nd, Sc) due to the chemical composition of Mg_{78.3}Nd_{19.4}Sc_{2.2}. The needle-like precipitates are observed inside the Mg_{x}(Mg, Nd, Sc) phase. The XRD pattern has demonstrated four phases in this as-cast alloy, i.e. bcc\_A2, hcp, Mg_{3}Nd, and an unindexed phase with fcc structure. We were unable to tell fcc from MgSc\_bcc via detecting the chemical composition of phases due to the limit of beam spot size in EPMA. The fcc and MgSc\_bcc phases have not been labeled in figure 11(a). Due to the bcc\_A2 phase is stable at high temperature in Mg-Sc rich alloys, we can infer that the 13\# alloy has crystallized into (Mg\_bcc) phase firstly, then the complex phase transformations during the furnace cooling have introduced the other phases. The verification and rationalization of the transformation pathways in this alloy are still open.

Figure 8. The inferred liquidus projection in comparison with the primary crystal observed from this experiment.
4. Conclusions

In summary, the phase equilibria in Mg–Nd–Sc system have been studied via analyzing phase constitution and phase chemical composition in annealed alloys. The isothermal sections at 500 °C, 530 °C, and 550 °C have been constructed by assessing all the experimental data in this work. Mg₄₁Nd₅ shows negligible ternary solubility in

Figure 9. The as-cast microstructure and XRD patterns of alloy samples 1# (a), (b) and 4# (c), (d).

Figure 10. (a) SEM back-scattered electron image and (b) XRD pattern of the as-cast alloy Mg₉₂.₆Nd₇.₂Sc₀.₂.
comparison with noticeable ternary solubility of Mg and Sc in Mg3Nd lattice. The structure homogeneity range of Mg3(Mg, Nd, Sc) phase slightly increases with the increase of temperature. The matrix phases of bcc and/or hcp together with the precipitations of Mg3(Mg, Nd, Sc) and other ordering structures provide an improved space for microstructure and composition modulation. The results of this work offer an overview of the phase diagram for composition design in the Sc alloyed Mg–Nd alloys.

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Conflict of interest statement

The authors state that they have no conflicts of interest in this work.
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