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ScienceDirect
Trans. Nonferrous Met. Soc. China 25(2015) 3877−3885
Transactions of Nonferrous Metals Society of China
www.tnmsc.cn

Effects of neodymium addition on microstructure and mechanical properties of near-eutectic Al−12Si alloys

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Received 8 February 2015; accepted 11 May 2015

Abstract: The microstructure and mechanical properties of near-eutectic Al−12Si alloys modified with 0−0.4% Nd (mass fraction) were investigated. The results indicate that a submicro- or nano-sized Al_2Nd phase is observed in the modified alloy with 0.3% Nd. The morphology of the α(Al) phase is significantly refined in the Nd-modified alloys. The primary Si morphology simultaneously changes into a fine, particle-like morphology, and the morphology of eutectic Si becomes fine-fibrous instead of coarse-acicular. Relatively few growth twins are observed on the surface of the Si plate in the Al−12Si−0.3Nd alloy at the optimal modification level. The mechanical property test results confirm that the mechanical properties of the as-cast Al−12Si alloys are enhanced after the Nd addition, with optimal ultimate tensile strength (UTS) of 252 MPa and elongation (EL) of 13% at an Nd content of 0.3%. The improved mechanical properties are attributed to the refined morphology of Si phase and the formation of the Al_2Nd phase.

Key words: Al−12Si alloy; neodymium; refinement; growth twins; mechanical properties

1 Introduction

Al−Si alloys with near-eutectic composition are commonly utilized in foundry applications, favored for their superior castability, balanced strength, ductility and corrosion resistance [1,2]. The α(Al) dendrite and Si phase characteristics of near-eutectic Al−Si alloys significantly influence the final mechanical properties of the casts and products [3]. The usual method of refining the microstructure of near-eutectic Al−Si alloys is the addition of certain modified element, though electromagnetic stirring, rapid solidification, and melt overheating treatment have also been used to modify the morphology of primary and eutectic Si [4−6].

Alloying with rare earth elements such as ytterbium (Yb) [7], strontium (Sr) [8], scandium (Sc) [9], samarium (Sm) [10] and erbium (Er) [11] has been shown to effectively improve the microstructure and tensile properties of near-eutectic Al−Si alloys. LIAO et al [12], for example, reported a considerable increase of the amount of α(Al) dendrites in Al−11.6%Si (mass fraction) alloys modified with Sr, which exhibited favorable mechanical properties and further confirmed that the mechanical properties of modified alloys are linearly related to the volume fraction of α(Al) dendrites. MAZAHERY and SHABANI [13] investigated the effect of secondary dendrites arm spacing (SDAS) values of α(Al) dendrites on the mechanical properties of the yttrium (Y)-modified cast Al−Si automotive Al alloys. The results showed that the strength of the Y-modified alloy declines with increasing the SDAS values. They also found that Sr addition reduces the average particle area and length of Si particle to a great extent [14]. Rare earth (RE) modifiers caused the morphologies of large, blocky primary Si and coarse, acicular eutectic Si in the Al−Si alloys to change into tiny, block-shaped and fine-fibrous morphologies [15,16]. Though the exact mechanism behind this process is still somewhat under debate, the impurity-induced twinning (IIT) mechanism is widely accepted. The IIT mechanism is defined by the adsorption of impurities on the growth surfaces of Si phases, which causes increased twinning. The ideal ratio of atomic radius of the modifier to Si ($r_{modifier}$:r_{Si}) to trigger IIT is about 1.646. Uniformly-dispersed and RE-rich particles with micro or nano size have been observed in Al−Si alloys modified with small amounts of rare earth elements [17,18]. However, the research on

Foundation item: Projects (51405216, 51165032) supported by the National Natural Science Foundation of China; Project (20151BAB216018) supported by the Natural Science Foundation of Jiangxi Province, China; Project (GJJ14200) supported by the Education Commission Foundation of Jiangxi Province, China
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DOI: 10.1016/S1003-6326(15)64035-3

the crystal structure of RE-rich particles and their specific strengthening mechanism in Al−Si alloys is yet limited.

According to the previous research [19,20], the Si phase morphology in Al−Si alloys was altered to some extent after pure Nd was added as a modifier, but the test results remained unsatisfactory. The specific alloying behavior of Nd in Al−Si alloys and the structural characteristics of Nd-rich intermetallic compounds are not fully understood. In effort to remedy this, the present study investigated the effects of Nd addition at different levels on the microstructures of α(Al) dendrites and Si phases, and the mechanical properties of near-eutectic Al−12Si (mass fraction, %) alloys, focusing particularly on the Nd-rich intermetallic compounds and effects of rare earth Nd on Si twinning.

2 Experimental

Al−12Si−xNd alloys (x=0, 0.1, 0.2, 0.3, 0.4) (mass fraction, %) were prepared from pure Al ingots, Al−20Si master alloy and Al−20Nd master alloy. Pure Al ingots and Al−20Si master alloy were melted in a graphite crucible at 760 °C by electric resistance furnace. After degassing and slagging-off, Al−Nd master alloy was added. In order to completely dissolve Nd, the molten samples were held at 680 °C for about 30 min and then poured into a mold at 700 °C. The chemical components of the specimens were analyzed by inductively-coupled plasma atomic emission spectroscopy (ICP-AES). The results are shown in Table 1.

Table 1 Chemical compositions of Al−12Si−xNd alloys (mass fraction, %)

Alloy	Si	Cu	Fe	Mn	Nd	Al
Al−12Si	12.01	0.26	0.62	0.46	−	Bal.
Al−12Si−0.1Nd	11.96	0.26	0.59	0.45	0.08	Bal.
Al−12Si−0.2Nd	11.97	0.27	0.65	0.45	0.21	Bal.
Al−12Si−0.3Nd	11.95	0.25	0.60	0.46	0.30	Bal.
Al−12Si−0.4Nd	11.99	0.27	0.62	0.45	0.39	Bal.

An etchant of 0.5% HF in alcohol solution was used to reveal the microstructures of the polished specimens. The microstructures of specimens were observed using an optical microscope (OM) (Nican M300). The precipitated phases and twins in Si of the Al−12Si−xNd alloys were characterized by X-ray diffraction (XRD) (Bruker D−8), scanning electron microscopy (SEM) (Quanta 200) equipped with energy dispersive spectroscopy (EDS) and transmission electron microscopy (TEM) (JEM−2100). The TEM foils were prepared by twin-jet polishing in an electrolyte of 5% (volume fraction) perchloric acid in alcohol.

The Nd modification's effects on the primary Si in Al−12Si alloys were quantified employing average size ($\overline{D}$) and volume fraction (φ_V), similarly to mean area ($\overline{A}$) and aspect ratio (R_A) which were used to characterize the modification of eutectic Si. The four following equations were calculated:

$$\overline{D}=\frac{1}{m}\sum_{j=1}^{m}\left(\frac{1}{n}\sum_{i=1}^{n}\sqrt{\frac{4A_{pi}}{\pi}}\right)_j \tag{1}$$

$$\varphi_V=\frac{1}{m}\sum_{j=1}^{m}\left[\left(\sum_{i=1}^{n}A_{pi}\right)_j\Big/A_j\right] \tag{2}$$

$$\overline{A}=\frac{1}{m}\sum_{j=1}^{m}\left(\frac{1}{n}\sum_{i=1}^{n}A_{ei}\right)_j \tag{3}$$

$$R_A=\frac{1}{m}\sum_{j=1}^{m}\left[\frac{1}{n}\sum_{i=1}^{n}\left(\frac{L_l}{L_s}\right)\right]_j \tag{4}$$

where A_{pi} is the area of a single primary Si particle, A_{ei} is the area of a single eutectic Si particle, A_j is the area of a single field, m is the number of fields, n is the number of particles in a single field, and L_l/L_s is the ratio of the longest to the shortest dimensions of a single eutectic Si particle. The parameters in these equations were measured with an Image-Pro Plus 6.0 image analyzer (IPP). The smaller the four values, the finer the primary and eutectic Si.

The as-cast samples were prepared for tensile property testing according to ASTM B557M specifications. Tensile property tests were performed on a material testing machine (UTM5105) at tension speed of 2.0 mm/min. There were four test samples in each group for each specific Nd content.

3 Results and discussion

3.1 Microstructure

The morphologies of α(Al) dendrites in the as-cast Al−12Si−xNd alloys are shown in Fig. 1. In the unmodified Al−12Si alloy (Fig. 1(a)), the coarse α(Al) phases contain many large, blocky primary Si, and coarse-acicular eutectic Si. When the Al−12Si alloy was modified with Nd, the morphology of the α(Al) phase changes from coarse-blocky into fine primary dendrite and secondary dendrite, as shown in Figs. 1(b)−(e). SDAS was used to analyze the refining behavior of the α(Al) phases by quantitatively measuring over 20 spatial fields in each specimen under IPP. The relationship between Nd content and SDAS of the alloys is shown in Fig. 2. As the Nd content increases from 0 to 0.4%, the SDAS values first decreases to 18 μm, and then slightly

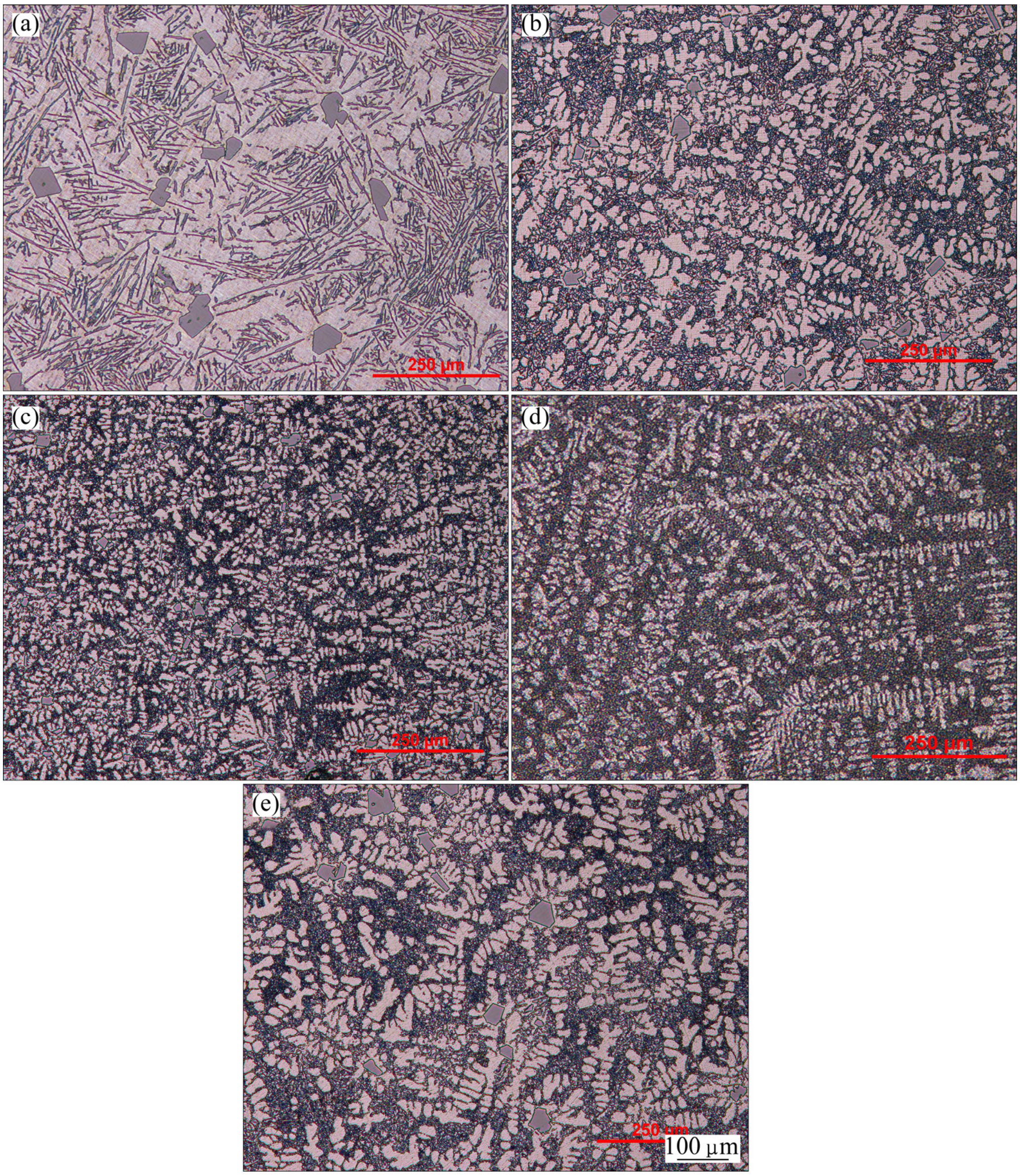

Fig. 1 Morphologies of α(Al) dendrites in as-cast Al−12Si−xNd alloys with differing Nd contents: (a) Al−12Si; (b) Al−12Si−0.1Nd; (c) Al−12Si−0.2Nd; (d) Al−12Si−0.3Nd; (e) Al−12Si−0.4Nd

increases up to 20 μm. The finest α(Al) dendrite is obtained at 0.3% Nd, producing the alloy with the smallest SDAS.

According to scaling law $\lambda_2=R/2$, the SDAS values of variations in λ_2 for small Peclet number conditions (such as metal systems, as described by TRIVEDI and SOMBOONSUK [21]) depend on the following equation:

$$\lambda_2=\left(8\Gamma DL\big/(kv\Delta T_0)\right)^{1/2} \tag{5}$$

where λ_2 is the SDAS value, Γ is the Gibbs Thomson coefficient, D is the diffusion coefficient in liquid, L is a constant depending on harmonic perturbations ranging from 10 to 28, k is the distribution coefficient, v is the

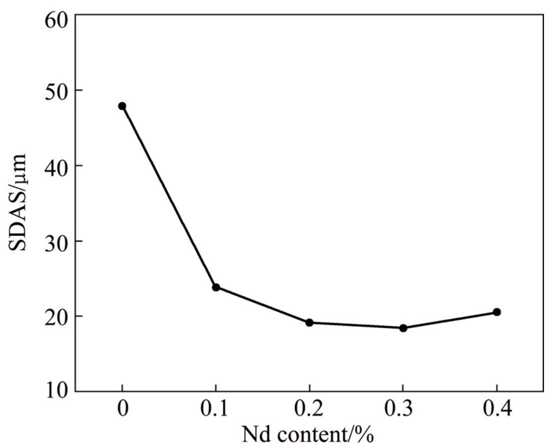

Fig. 2 Relationship between Nd content and SDAS of as-cast Al−12Si alloys

dendritic front growth rate, and ΔT_0 is the difference between liquid and solid equilibrium temperatures. The stable Al_2Nd phases with high melting point are rich in liquid before the α(Al) dendritic growth frontier, which causes the composition undercooling and increases the ΔT_0 value, which then decreases the λ_2. As a result, the λ_2 values of alloys modified with Nd are consistently less than half of λ_2 value for the unmodified alloy.

Figure 3 shows the XRD patterns of the as-cast Al−12Si alloy and the as-cast Al−12Si−0.3Nd alloy. Only the α(Al) and Si phases show peaks in the Al−12Si alloy and Al−12Si−0.3Nd alloy, as shown in Fig. 3. In Fig. 3(b), no Nd peak phase is observed in the modified alloy, possibly due to the fact that the Nd-containing phase is too small to be detected by XRD.

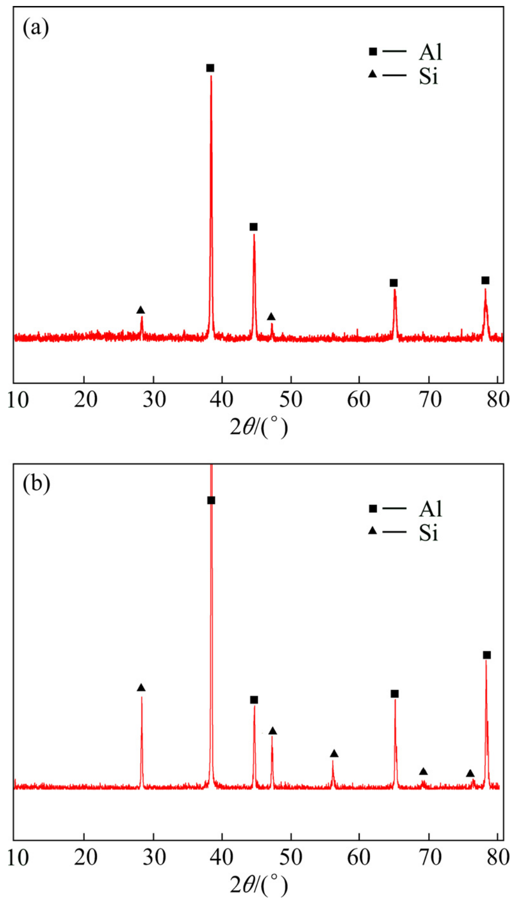

Fig. 3 XRD patterns of as-cast Al−12Si alloy (a) and Al−12Si−0.3Nd alloy (b)

Figure 4 presents the SEM image and EDS analysis results of the as-cast Al−12Si−0.3Nd alloy. Many white particles are distributed uniformly in the modified alloy, as shown in Fig. 4(a). Three white particles were measured by digital image measurement software IPP, yielding diameters of 0.366, 0.275, and 0.084 μm. As shown in Fig. 4(b), according to the EDS results, the white particle is comprised of Al, Si and Nd, with the atomic ratio of about 83:16:0.15, suggesting that the particle is an Nd-rich intermetallic compound in the Al−12Si alloy modified with Nd. The TEM analysis further characterizes the Nd-rich intermetallic compound, as shown in Fig. 5. Figure 5 shows the TEM image of the as-cast Al−12Si−0.3Nd alloy, where Nd-rich phases are observable. The corresponding selected area diffraction pattern (SADP) for Nd-rich phase, which confirms that the Nd-rich phase is an Al_2Nd phase (face centered cubic structure, a=0.8 nm [22]), is provided in the inset of Fig. 5.

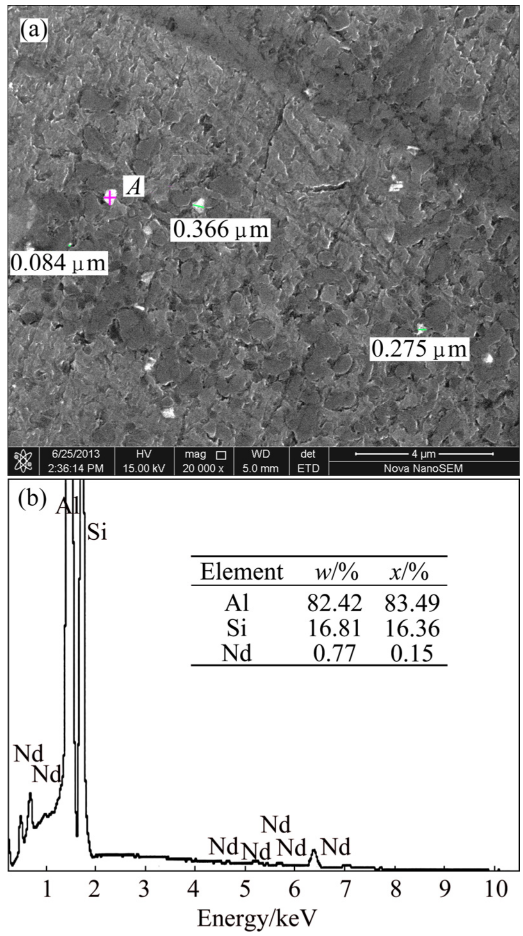

Fig. 4 SEM image (a) and EDS analysis of point A (b) of as-cast Al−12Si−0.3Nd alloys

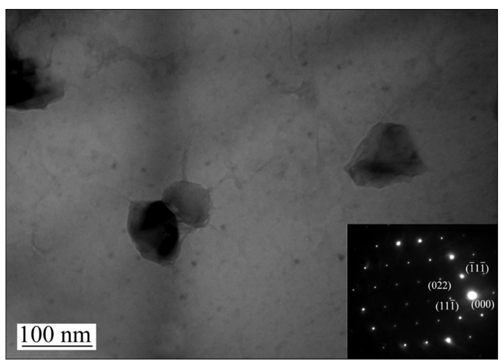

Fig. 5 TEM image of Al_2Nd phase in Al−12Si−0.3Nd alloy and [011] diffraction pattern of Al_2Nd phase

Figure 6 shows the Si phase morphologies of Al−12Si alloys with varying Nd contents. The Si phase morphology in unmodified alloy presents large, blocky primary Si with sharp corners, and coarse-acicular eutectic Si, as shown in Fig. 6(a). The addition of modifier Nd refines the primary and eutectic Si considerably, as shown in Figs. 6(b)−(e). As the Nd content increases, the primary Si morphology changes from large, blocky to fine and particle-like, and some primary Si phases are fully modified and eventually disappear. The coarse-acicular eutectic Si phases become short rods in the Al−12Si alloy modified with 0.1% Nd. The further addition of Nd, up to 0.2% and 0.3%, transforms the eutectic Si from short-rod to fine-fibrous (Figs. 6(c) and (d)). As the Nd content continues to increase, the modifying effect of Nd on the eutectic Si in the Al−12Si−0.4Nd alloy weakens, as shown in Fig. 6(e).

Figure 7 depicts quantitative metallographic results of Si phases in the Al−12Si−*x*Nd alloys. The average diameter and volume fraction of primary Si decrease considerably after the addition of Nd. When the Nd content exceeds 0.2%, the changes in average diameter and volume fraction of primary Si are quite small (Fig. 7(a)), and the alloy is at the minimum average diameter (10.8 μm) and volume fraction (0.34%). When the Nd content increases to 0.3%, the mean area and aspect ratio of eutectic Si decrease from 88.7 μm^2 and 3.0 (unmodified alloy) to 17.8 μm^2 and 1.9, respectively. The mean area and the aspect ratio of eutectic Si increases, however, with the Nd addition over 0.3%, as shown in Fig. 7(b). These results suggest that Nd has a remarkable impact on both the average diameter and volume fraction of primary Si, and the mean area and aspect ratio of eutectic Si. The 0.3% Nd-modified alloy presents finer primary Si and eutectic Si than alloys with 0, 0.1%, 0.2%, and 0.4% Nd.

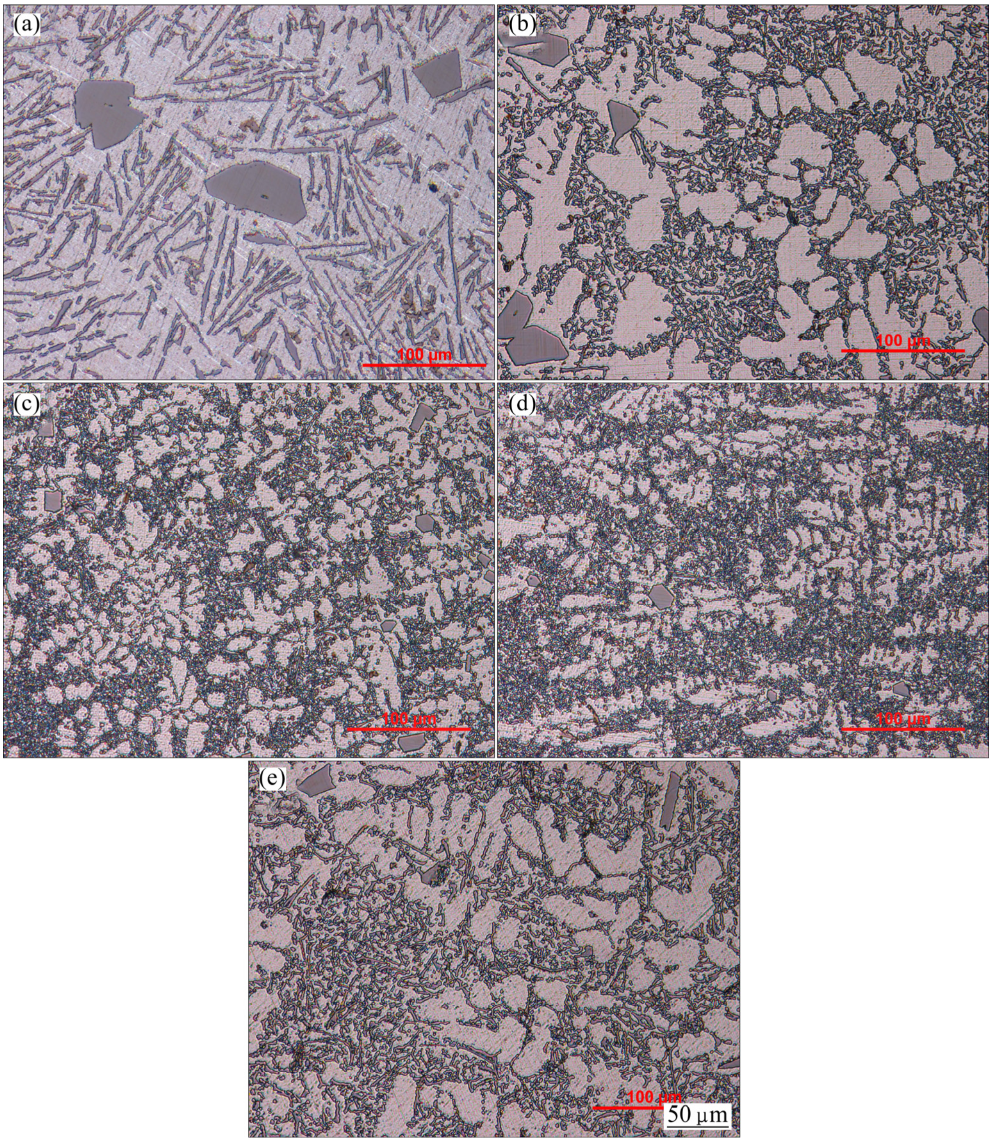

Fig. 6 Si phase morphologies of Al−12Si alloys with varying Nd contents: (a) Al−12Si; (b) Al−12Si−0.1Nd; (c) Al−12Si−0.2Nd; (d) Al−12Si−0.3Nd; (e) Al−12Si−0.4Nd

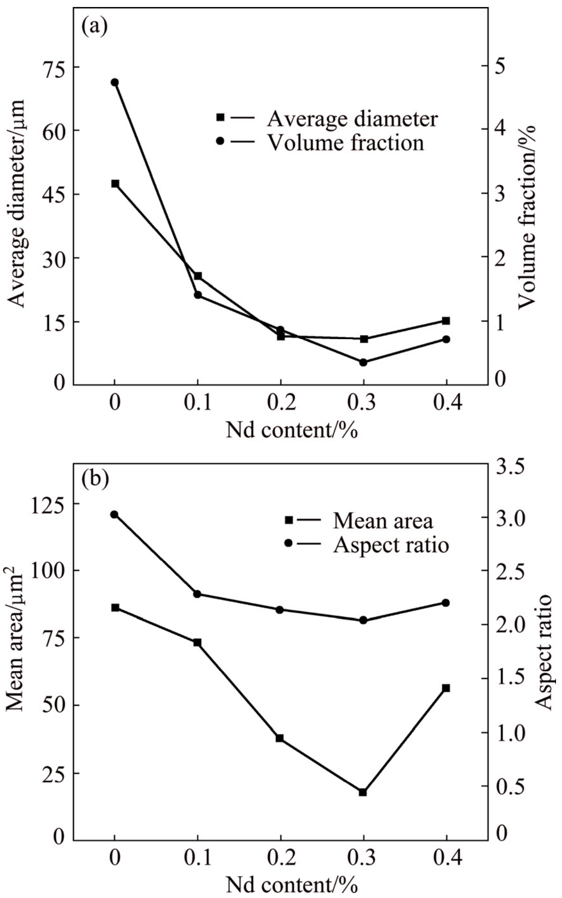

Fig. 7 Image analysis of primary Si (a) and eutectic Si (b) in Al−12Si−xNd alloys with differed Nd additions

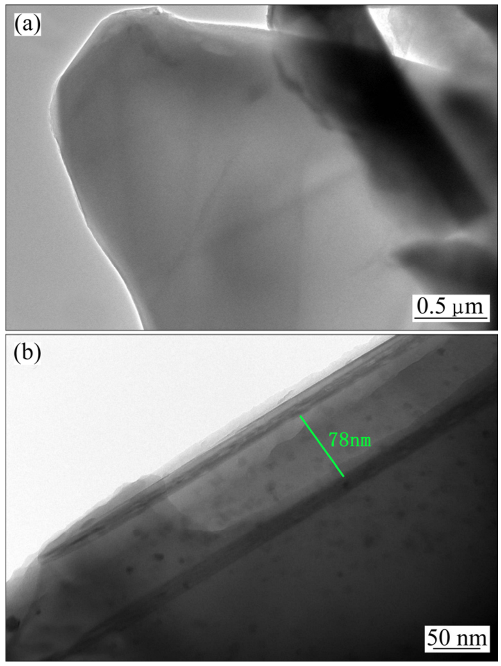

Fig. 8 TEM images of twins in Si phases of as-cast Al−12Si alloy (a) and Al−12Si−0.3Nd alloy (b)

Many previous studies centered around Al−Si alloys have investigated the mechanism of the Si phase modification. Based on the twin plane reentrant edge (TPRE) mechanism, the IIT model is widely accepted as the mechanism that facilitates the Si modification in the Al−Si alloys. According to IIT, the atoms of modifier are absorbed systematically on the solid−liquid interface. Because the size of modifier is larger than that of Si, Si atoms are not able to attach to their respective crystallographic sites, and thus the growth of the Si phase is hindered along the preferential direction. This changes the growth direction of the Si phase, and promotes the formation of twins [23]. The twins in Si phases of the unmodified alloy and the Al−12Si−0.3Nd alloys were analyzed by TEM in order to investigate the rare earth Nd's effects on the Si twinning. Figure 8(a) shows the Si phase surface of the unmodified alloy, which has no obvious twins. In the modified alloys, a few growth twins spaced about 78 nm apart on the surface of the Si plate are observed, as shown in Fig. 8(b).

In the previous study, NOGITA et al [24] evaluated the Si twinning in hypoeutectic Al−Si alloys with the additions of barium (Ba), calcium (Ca), yttrium (Y) and ytterbium (Yb), and elements were chosen due to their nearly ideal radio for twinning. The average twin spacing of Ba, Ca, Y and Yb-modified eutectic Si (from 200 to 500 nm) was significantly lower than that of unmodified alloy, but higher than that of Sr-modified alloy (26 nm) [25]. They considered the twin densities of all modified samples to be lower than the expected values after making predictions based on the IIT model. CHANG [26] asserted that only a slight increase of eutectic Si twin density was found in RE-modified Al−Si alloy, which was agreement with the hypothesized results. The twin probability of 1% RE (mass fraction) modified alloys was well below that of Sr-modified alloy, at 0.0031 versus 0.0245 [27]. Considering the results shown in Fig. 8(b) and the results of the previous research, the change in twin density alone does not sufficiently explain the modification effects of RE addition on the Al−Si alloys.

Although RE supplementation's effects are still relatively unknown, the Nd modification of primary Si and eutectic Si is clearly visible, as shown in Figs. 6(b)−(e). To this effect, the change in Si phase growth patterns is considered to be the primary cause of structural modification in Al−Si alloys modified with Nd or RE.

3.2 Mechanical properties and fracture

The ultimate tensile strength (UTS) and elongation (EL) of Al−12Si−*x*Nd alloys at room temperature are shown in Fig. 9. The UTS and EL values both increase first when Nd is added from 0 to 0.3%, and then decrease as the Nd content reaches 0.4%. Compared with the UTS and EL values of Al−12Si alloy (162 MPa and 4.2%), Al−12Si alloys modified with Nd show better mechanical properties, with an optimal UTS value of 252 MPa and EL of 13%, at the Nd content of 0.3%. The corresponding UTS and EL values of Al−12Si alloys with the additions of 0.1%, 0.2% and 0.4% Nd are 193 MPa and 6.7%, 235 MPa and 10.8%, and 226 MPa and 9.4%, respectively.

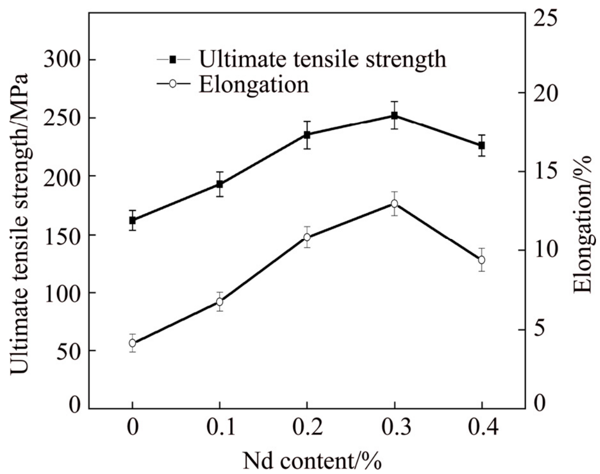

Fig. 9 Mechanical properties of as-cast Al−12Si−*x*Nd alloys at room temperature

It has been confirmed that the mechanical properties of as-cast Al−Si alloys depended primarily on the characteristics of *α*(Al) dendrites and Si phase, such as size, morphology and distribution, as reported by LI et al [28]. As discussed above, refined *α*(Al) dendrites and Si phases in the alloys modified with Nd confirm that the Nd element influences the mechanical properties of modified alloys. The enhancement of mechanical properties caused by the Nd addition is explained best from the following two perspectives.

1) Si phase refinement: Large, blocky primary Si with sharp corners, and coarse-acicular eutectic Si, considerably weaken the mechanical properties of Al−Si alloy. During the deformation process, the sharp corners and coarse-acicular structure of the Si phase can fragment the *α*(Al) matrix, causing rapid material failure. When Nd modifier is added to the Al−12Si alloys, large, blocky primary Si becomes fine and particle-like. The eutectic Si transforms at the same time from coarse-acicular to fine-fibrous, as shown in Figs. 6(b)−(e). The changes in Si phase characteristics result in weaker separating effect between the Si phase and alloy matrix, thus deteriorating mechanical properties in the alloys.

2) Al_2Nd phase strengthening. After Nd is added to the alloy, homogeneous, submicron or nano-sized Al_2Nd forms during the solidification process, as shown in Fig. 2. The Al_2Nd particles are coherent with the matrix, and thus effectively hinder dislocation and securely pin the grain boundary, altogether improving the mechanical properties of the modified alloy.

Notably, as shown in Fig. 9, the mechanical properties decline slightly once the Nd content exceeds 0.3%. At this point, the size and amount of Al_2Nd have become excessive in the Al−12Si−0.4Nd alloy and grow too large compared with that in the Al−12Si−0.3Nd alloy. Over the course of the solidification process, most of Nd atoms are pushed to the solid liquid interface and react with Al atoms to form Al_2Nd phases. When the Nd content is high enough in the modified alloys, a portion of the Al_2Nd particles form cluster morphology [29]. Once the cluster area reaches a specific level, the stress concentration causes the alloys to fracture quite readily during the deformation process.

The fracture morphologies of the as-cast Al−12Si alloys with different Nd contents observed by SEM are shown in Fig. 10. The fractograph of the Al−12Si alloy shows plenty of cleavage planes and only few tearing ridges on the fracture surfaces, indicating that the fracture form of the unmodified alloy is ductile and brittle mixed-fracture. The tearing ridges and tiny dimples significantly increase once Nd is added, as shown in Figs. 10(b)−(e). As shown in Figs. 10(c) and (d), plenty of tearing ridges and tiny dimples are present on the fracture surfaces of the alloys containing 0.2% Nd, and 0.3% Nd, indicating a ductile fracture mechanism.

All in all, it is reasonable to conclude that Al−12Si alloys supplemented with Nd show superior mechanical properties compared with the alloy that does not contain Nd. Among all the modified alloy samples, the one with the largest amount of tearing ridges and dimples is Al−12Si−0.3Nd, which also shows the optimal UTS value of 252 MPa and EL of 13%.

4 Conclusions

1) The Nd addition to the as-cast near-eutectic Al−12Si alloys results in highly refined *α*(Al) phase morphology. Submicro- or nano-size Nd-rich intermetallic compounds in Al−12Si alloys modified with Nd addition are confirmed, forming Al_2Nd phases with face-centered cubic structure. The primary Si morphology becomes fine and particle-like, and the eutectic Si phases transform from coarse-acicular to fine and fibrous. A few growth twins on the surface of the Si plate are observed, implying that the IIT mechanism does not effectively explain the modification effect of Nd addition on the Al−Si alloys.

2) The Al−12Si alloys modified with Nd, when compared with Al−12Si alloy (UTS of 162 MPa and EL of 4.2%), show better mechanical properties with optimal values of 252 MPa for UTS and 13% for EL at 0.3% Nd

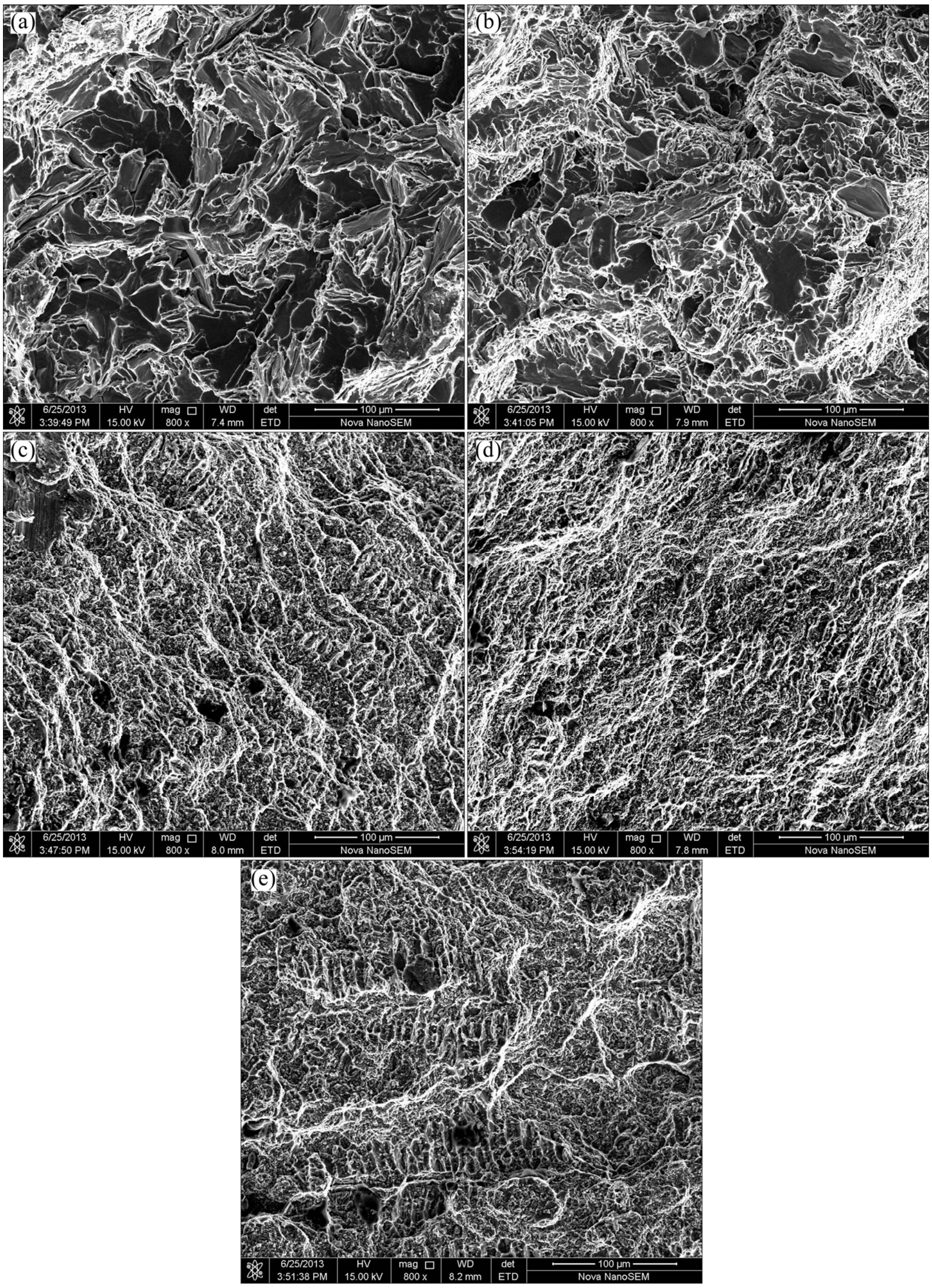

Fig. 10 Fracture morphologies of Al−12Si−*x*Nd alloys: (a) Al−12Si; (b) Al−12Si−0.1Nd; (c) Al−12Si−0.2Nd; (d) Al−12Si−0.3Nd; (e) Al−12Si−0.4Nd

supplementation. The improved mechanical properties are primarily attributed to the Si phase refinement and Al_2Nd phase strengthening.

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稀土元素 Nd 对近共晶 Al−12Si 合金显微组织与力学性能的影响

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摘　要：研究稀土元素 Nd(0~0.4%，质量分数)对近共晶 Al−12Si 合金显微组织与力学性能的影响。结果表明：在 0.3%Nd 改性的 Al−12Si 合金中形成一种亚微米或纳米尺寸的 Al_2Nd 相。在 Al−12Si 合金中添加稀土元素 Nd 能显著细化合金中的 α(Al)相，粗生硅相转变为细小颗粒状，共晶硅由粗大针状变成细小纤维状。在改性效果最佳的 Al−12−0.3Nd 合金的 Si 相表面观察到少量的生长孪晶。力学性能测试结果表明：添加 Nd 元素后，Al−12Si 合金的力学性能得到改善，当合金中 Nd 元素含量达到 0.3%时，合金的力学性能达到最优，抗拉强度(UTS)为 252 MPa，伸长率(EL)为 13%。合金力学性能的改善主要归因于合金中 Si 相形貌的改善和细小 Al_2Nd 相颗粒的形成。

关键词：AL−12Si 合金；钕；细化；生长孪晶；力学性能

(Edited by Mu-lan QIN)