

[Article ID] 1003- 6326(2001) 02- 0278- 03

Isothermal section (500 °C) of phase diagram of Nd-Al-Si ternary system^①

LONG Zhi-ling(龙志林)¹, ZHOU Yi-chun(周益春)¹, ZHUANG Ying-hong(庄应烘)²,
CHEN Rong-zhen(陈荣贞)², LIU Jing-qi(刘敬旗)²

(1. Institute of Fundamental Mechanics and Materials Engineering, Department of Physics,
Xiangtan University, Xiangtan 411105, P. R. China;

2. Industrial Testing and Experimental Centre, Guangxi University, Nanning 530004, P. R. China)

[Abstract] The isothermal section of the phase diagram of the ternary system Nd-Al-Si at 500 °C (Nd ≤ 50%, mole fraction) has been constructed on the basis of the data obtained by X-ray diffraction analysis, differential thermal analysis, metallographic examination, chemical analysis and electron microprobe analysis. The obtained diagram consists of 11 single-phase regions, 21 two-phase regions and 11 three-phase regions. There exist two limit solid solutions. The intermetallic compound NdAl_{1.5}Si_{0.5} has not been found in this section. No evidence of new phase has been observed in this work.

[Key words] Nd-Al-Si phase diagram; ternary section; neodymium aluminum silicides

[CLC number] TG 113.14

[Document code] A

1 INTRODUCTION

The Al-Si equilibrium phase diagram has been studied by many researchers^[1~5]. No intermetallic compound was discovered in Al-Si binary system. The solubility of Si in Al is 0.80% (0.77%, mole fraction), Al is insoluble in Si at 500 °C^[1]. However, Elliott^[2] reported that the solubility of Al in Si was 0.4% (mole fraction) at 577 °C (eutectic). The Nd-Al binary system was also studied^[6~9]. Six intermetallic compounds were observed, i. e. Nd₃Al, Nd₂Al, NdAl, NdAl₂, NdAl₃ and NdAl₄^[5,6]. After a re-investigation of this system, Buschow et al^[8] confirmed that Al-rich compound NdAl₄ was shown to be Nd₃Al₁₁. This compound forms peritectically at 1235 °C from the melt and NdAl₂ exists in two allotropic forms, with a transition at 950 °C (α -Nd₃Al₁₁ \leftrightarrow β -Nd₃Al₁₁)^[8], α -Nd₃Al₁₁ has orthorhombic structure, whereas β -Nd₃Al₁₁ has tetragonal structure of Al-deficient Al₄B₅-type crystal structure and lattice parameter data for Nd-Al phases have been recorded^[9]. Previous work^[10~13] has demonstrated that there exist six intermetallic compounds in the Nd-Si binary system, such as Nd₅Si₃, Nd₅Si₄, NdSi, Nd₃Si₄, Nd₂Si₃ and NdSi_x. The intermetallic compound information of the crystal structure and crystallography were reported^[10]. No investigation of the Nd-Al-Si ternary system has been reported.

2 EXPERIMENTAL

The neodymium, aluminum and silicon used in

this work were of 99.9%, 99.99% and 99.999% in purity, respectively. The alloy billets were prepared in an argon atmosphere in a high induction furnace or a vacuum arc furnace. All samples were melted in Al₂O₃ crucibles. The melting time was 3~5 min, which is long enough to obtain ingots with smooth surface and uniform compositions. In all, 212 samples having mass of 3g each were prepared. The composition of alloys did not change significantly because less than 0.5% of the total mass was lost in each synthesis.

The samples were kept sealed in silica tubes in vacuum during homogenization. The homogenization temperature of the alloys were chosen on the basis of the binary phase diagrams of the binary Nd-Al, Nd-Si and Al-Si systems and differential thermal analysis of some ternary alloy samples. Alloy was homogenized for 90~140 d at 520~750 °C. The alloys containing more than 20% (mole fraction) of Nd were diffusely annealed at 750 °C for 90 d; the alloys containing 10%~20% (mole fraction) of Nd were annealed at 600 °C for 110 d; the alloys containing less than 10% (mole fraction) of Nd were annealed at 520 °C for 140 d. Then all the alloys were furnace cooled at a rate of about 3 °C·h⁻¹ to 500 °C, kept for 7 d and then quenched into an ice-water mixture. The alloy powders were obtained by hammering of brittle ingots or chiselling of ductile ingots. The powders of ductile ingots were sealed in small glass tubes in vacuum and annealed at 500 °C for 10 d, followed by liquid nitrogen quenching. The powders were not stress-relieved. Diffraction patterns of all alloys are very clear with a weak back ground.

① **[Foundation item]** Project (10072052) supported by the National Natural Science Foundation of China

[Received date] 2000- 9- 18; **[Accepted date]** 2000- 11- 22

The X-ray diffraction analysis was performed with powder samples using a Rigaku 3015 diffractometer and a Debye-Scherrer camera with a diameter of 114.6 mm. Copper targets and nickel filters were used. Differential thermal analysis was carried out in a CR-G type analyzer at the rate of $10\text{ }^{\circ}\text{C}\cdot\text{min}^{-1}$ or less. Alloy compositions were determined by electron probe microanalysis, using an EPM-810Q instrument. Metallographic examinations were done using a Hitachi S-570 type scanning electron microscopy. The thermal treatment of alloys was carried out in an electronic resistance furnace in quartz ampoule. The temperature was controlled within $\pm 5\text{ }^{\circ}\text{C}$. Metallographic samples of alloys were etched using a solution of $\text{HNO}_3 + \text{HCl} + \text{H}_3\text{PO}_4 + \text{HAc}$ mixture acid.

3 RESULTS AND DISCUSSION

3.1 Binary system

The mutual solid solubility limits of Al and Si were determined by means of lattice parameter measurements, electron micro-probe analysis and metallographic examinations. The results are that the maximum solid solubility of Si in Al is 0.82% (mole fraction), Si in Al does not indicate any detectable solid solubility at 500 °C. The data coincide satisfactorily with the known information on this system^[1,2], since we used high-purity Al and Si and annealing times are long enough to ensure equilibrium.

There exist four intermetallic compounds in the binary Nd-Al system ($\text{Nd} \leq 50\%$, mole fraction)^[8,9]. We also studied this system and confirmed that the four intermetallic phases exist at 500 °C, i. e. NdAl, NdAl₂, NdAl₃ and $\alpha\text{-Nd}_3\text{Al}_{11}$, the solid solubility of Nd in Al is too small to be observed, in good agreement with Buschow et al^[8].

In the binary Nd-Si system, two intermetallic compounds, Nd₂Si₃ (or NdSi_{1.5}) and NdSi_x were reported ($\text{Nd} \leq 40\%$, mole fraction) by Eremenko^[11], Dvorina^[12] and Raman^[13]. Eremenko thought that Nd₂Si₃ may be expressed as NdSi_{1.5}. Many researchers assumed that NdSi_x has rather wide homogeneity range. We confirmed that Nd₂Si₃ and NdSi_x existed. Nd₂Si₃ is observed for its low-temperature modification $\alpha\text{-Nd}_2\text{Si}_3$. The other compound NdSi_x exists in $\beta\text{-NdSi}_x$ and has a homogeneity range extending from about 64% (mole fraction) Si to 67% (mole fraction) Si. Our experimental results show agreement with those of Eremenko^[10] and Dvorina^[12].

3.2 Ternary system

SEM microstructures were shown in Fig. 1. Some information on the intermetallic compounds in ternary Nd-Al-Si system was given by Gschneidner et al^[14]: NdAl_{1.25}Si_{0.75}, NdAl_{1.5}Si_{0.5}, NdAl_{1.75}Si_{0.25}

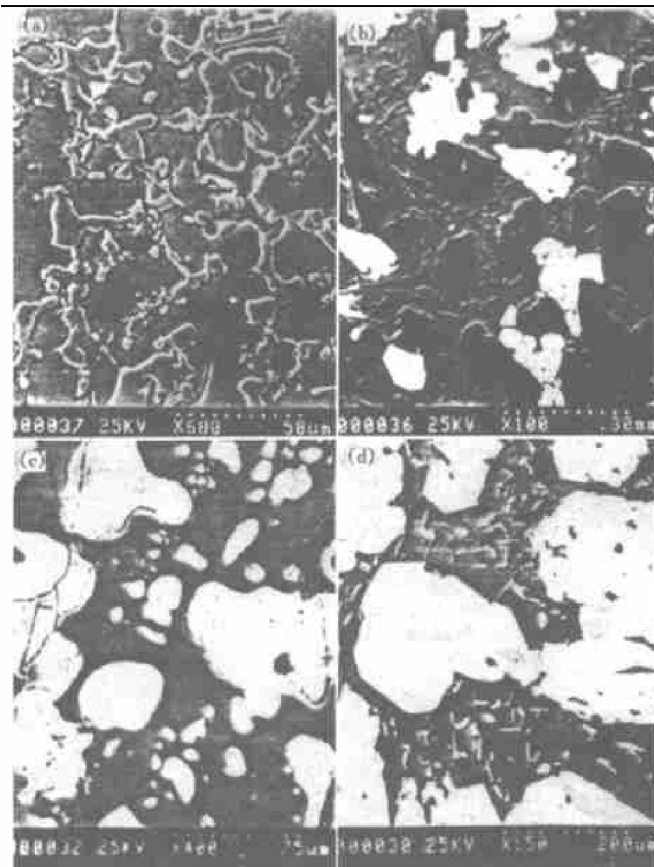


Fig. 1 Microstructures of Al-Nd-Si ternary alloys at 500 °C

- (a) —Sample 212 (Nd 33.3% (mole fraction) Si 16.7% (mole fraction) Al alloy) NdAl_{1+x}Si_{1-x} + NdAl_{1.75}Si_{0.25};
- (b) —Sample 170 (Nd 6% (mole fraction) Si 50% (mole fraction) Al alloy) NdAl₂Si₂ + Al + Si;
- (c) —Sample 150 (Nd 35% (mole fraction) Si 33% (mole fraction) Al alloy) $\beta\text{-NdSi}_x$ + NdAl_{1+x}Si_{1-x};
- (d) —Sample 142 (Nd 23% (mole fraction) Si 1% (mole fraction) Al alloy) NdAl_{1+x}Si_{1-x} + NdAl₃ + $\alpha\text{-Nd}_3\text{Al}_{11}$

and NdAl₂Si₂. The following three compounds were verified: NdAl₂Si₂, NdAl_{1.75}Si_{0.25} and NdAl_{1.25}Si_{0.75}. The compound NdAl_{1.25}Si_{0.75} has a comparatively wide range in composition and it seems to change rather continuously in its diffraction pattern as the composition changed, it may be expressed as NdAl_{1+x}Si_{1-x}, with $0 \leq x \leq 0.4$ ^[15]. By analyzing diffraction patterns of NdAl_{1.5}Si_{0.5}, NdAl_{1.25}Si_{0.75} and NdAl_{1.75}Si_{0.25} phases, it is found that the alloy with the composition of NdAl_{1.5}Si_{0.5} was composed of two phases: NdAl_{1.25}Si_{0.75} + NdAl_{1.75}Si_{0.25}, its microscopic structure is shown in Fig. 1(a). The phase $\beta\text{-NdSi}_x$ has a ternary homogeneity ranges. The maximum solubility of Al in $\beta\text{-NdSi}_x$ is about 1.5% (mole fraction).

3.3 Isothermal section

By comparing and analyzing the X-ray diffraction patterns of 212 samples and by identifying the phase in each samples, Fig. 2 shows the isothermal section of the phase diagram of the ternary system Nd-Al-Si

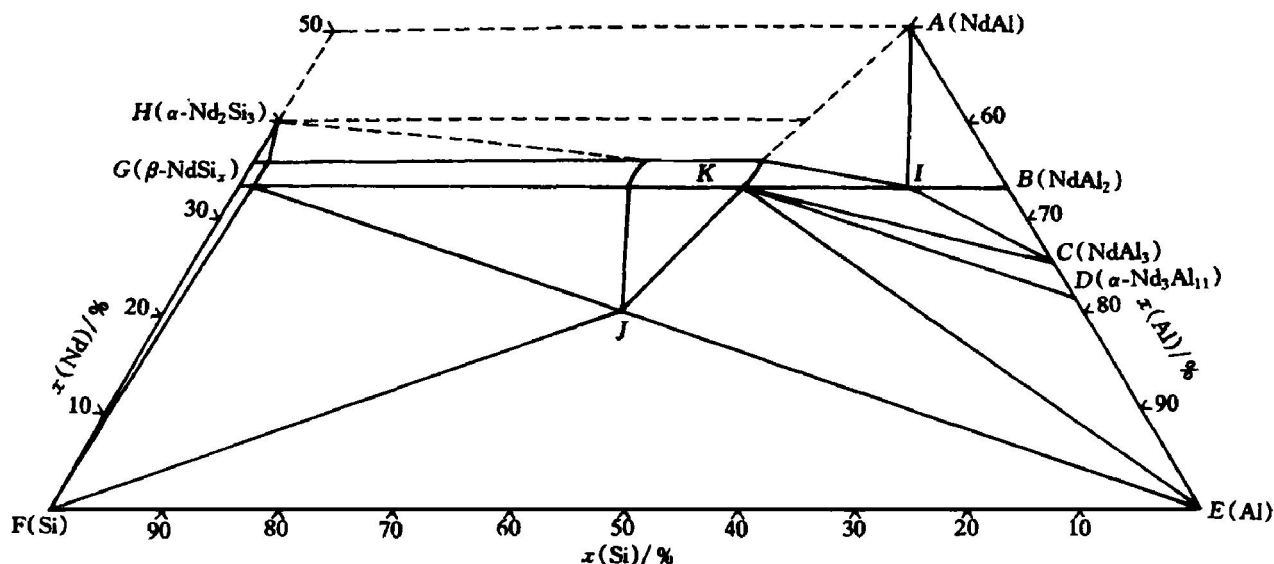


Fig. 2 Isothermal section of phase diagram of Nd-Al-Si ternary system at 500 °C

(Nd \leq 50%, mole fraction) has been determined at 500 °C. The section consists of 11 single-phase regions, 21 two-phase regions and 11 three-phase regions. The single-phase regions are A (NdAl), B (NdAl₂), C (NdAl₃), D (α -Nd₃Al₁₁), E (Al), F (Si), G (β -NdSi_x), H (α -Nd₂Si₃), I (NdAl_{1.75}Si_{0.25}), J (NdAl₂Si₂) and K (NdAl_{1+x}Si_{1-x}, 0 \leq x \leq 0.4); the two-phase regions are A + B, B + C, C + D, D + E, E + F, F + G, G + H, I + A, I + B, I + C, I + K, J + E, J + F, J + G, J + K, K + A, K + C, K + D, K + E, K + G and K + H; the three-phase regions are A + B + I, A + I + K, B + C + I, I + C + K, C + D + K, K + D + E, E + J + K, E + F + J, F + G + J, G + J + K and G + H + K.

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(Edited by HUANG Jin-song)