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Isothermal section of the Al–Dy–Ge ternary system at 673 K

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article info

ABSTRACT

Article history: Received 23 December 2009 Received in revised form 13 January 2011 Accepted 15 January 2011 Available online 22 January 2011

Keywords: Rare earth compound Crystal structure Phase diagram X-ray powder diffraction The isothermal section of the phase diagram of the Al–Dy–Ge ternary system at 673 K has been investigated by X-ray powder diffraction and scanning electron microscope equipped with energy dispersive X-ray spectroscopy in backscattered electron imaging modes. The existence of thirteen binary compounds including Dy_3Ge_4 in the system Dy–Ge has been confirmed. Four ternary compounds, namely AlDyGe, $Al_3Dy_2Ge_4$, $AlDy_2Ge_3$, and $AlDy_2Ge_2$ were observed, and five new ternary compounds, i.e., $\text{Al}_{0.33}\text{DyGe}_2$, AlDy₂Ge₆, Al₂DyGe₂, AlDy₃Ge₃, and Al_{3−x}Dy₁₁Ge_{7+x} (x ≤ 0.7), and one pseudo-binary compound $Al_{3-x}DyGe_x$ were found in this system at 673 K. The maximum solid solubility of Ge in the pseudo-binary compounds $Al_{3-x}DyGe_x$ is about 7.5 at.% with x = 0.3 at 673 K.

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1. Introduction

Al-based amorphous alloys have attracted considerable attention in recent years due to their outstanding mechanical and physical properties, such as high mechanical strength, good corrosion resistance, and unique magnetic properties. Especially they are attractive candidates for advanced high-strength lightweight materials [\[1,2\]. A](#page-9-0)s an important technological application in materials, rare earths have been commonly recognized as favorable additives because they have good effects on corrosion resistance, tensile strength and heat resistance of alloys [\[3\]. A](#page-9-0)ttentions are also paid to Germanium using as an addition to Aluminum alloys. Aluminum mechanically alloyed with Germanium exhibits improved super plastic elongation [\[4\].](#page-9-0) Properties of these alloys are strongly related with the formation of the intermetallic compounds. Therefore, it is necessary to investigate the phase diagram and thermodynamic description of RE–Al–Ge systems in order to develop alloys with required properties. At present, investigation on the phase relations for the RE–Al–Ge systems have not been investigated except the RE–Al–Ge (RE = Y, La, Ce, Sm, Tb) [\[5–9\]](#page-9-0) ternary systems, which have been determined only in the region with the compositions in the range of 0–40 at.% RE.

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A total of seven type intermetallic compounds in the RE–Al–Ge systems have been reported in references, i.e., REAlGe (RE=Y, La–Nd, Sm, Eu–Er, Lu) [\[10\],](#page-9-0) $RE_2Al_3Ge_4$ ($RE=La-Nd$, Sm, $Gd-Er$) [\[10\]](#page-9-0) RE_2AlGe_6 (RE = La–Nd, Sm, Gd) [10], $REAl_2Ge_2$ (RE = Y, La, Ce, Eu, Gd, Er, Yb) $[10-13]$ RE₂AlGe₃ (RE = Y, La, Nd, Sm, Tb, Dy, Ho, Er, Tm) [\[10,14–16\],](#page-9-0) REAl_{3−x}Ge_x (RE = Gd, Tb, Ho, X = 0.10–0.30) [\[17\], R](#page-10-0)EAl_xGe₂ (RE = Y, Gd, Dy, Ho, Er, $X = 0.27 - 0.35$) [\[18–22\], a](#page-10-0)nd $RE₂AlGe₂$ (RE = Gd, Dy) [\[23,24\].](#page-10-0)

The phase diagrams of the binary systems Al–Dy, Dy–Ge and Al–Ge in the Al–Dy–Ge system have been presented in Ref. [\[25\].](#page-10-0) At 673 K, there are five intermetallic phases $(A₁₃Dy, A₁₃Dy, A₂₃Dy, A₃₄Dy, A₄₅Dy, A₅₅Dy, A₆₅Dy, A₇₅Dy, A₈₅Dy, A₉₅Dy, A₁₅Dy, A₁₅Dy, A₁₅Dy, A₁₅Dy, A₁₅D$ Al₂Dy, AlDy, Al₂Dy₃ and AlDy₂) reported in the Al–Dy system [\[25–28\].](#page-10-0) Seven intermetallic phases, i.e., Dy_5Ge_3 , Dy_5Ge_4 , $DyGe_5$ Dy_2Ge_3 , Dy_3Ge_5 , $DyGe_2$ and $DyGe_3$ were found in the $Dy-Ge_3$ system [\[25,26\].](#page-10-0) The Al–Ge system [\[25\]](#page-10-0) is a simple eutectic system with three phases of the liquid, the Al-fcc solid solution and the Ge-diamond cubic solid solution. No intermetallic phase exists in the Al–Ge system. In this paper we report the investigations on the phase relations in the Al–Dy–Ge ternary system at 673 K.

2. Experimental details

The ingots of aluminum (99.99 wt.%), dysprosium (99. 8 wt.%) and germanium (99.99 wt.%) were used as starting materials. Samples were prepared by arc melting on a water-cooled copper crucible with a non-consumable tungsten electrode under high pure argon atmosphere. All samples were remelted three times and turned around after melting for better homogeneity. Weight

^{0925-8388/\$ –} see front matter © 2011 Elsevier B.V. All rights reserved. doi:[10.1016/j.jallcom.2011.01.080](dx.doi.org/10.1016/j.jallcom.2011.01.080)

Fig. 1. XRD patterns of (a) the sample No. 21 (Al₈₀Dy₂₀) contained the phases of Al₃Dy and Al and (b) the sample No. 56 (Al₅₅Dy₂₅Ge₂₀) located in the phase region of $AIDyGe + Al₃Dy + Al.$

losses of alloy buttons during arc melting were less than 1% of the total mass. All alloy samples after melting were subjected to a homogenizing annealing in evacuated quartz. The homogenization temperatures were chosen on the basis of the binary phase diagrams of the Al–Dy, Dy–Ge, and Al–Ge systems. Samples with compositions less than 20 at.% Dy were annealed at 673 K for 600 h. The Al-rich and Dy-rich samples were annealed at 873 K and 1173 K for 480 h, respectively. Samples with composition in the range of 30–70 at.% Ge were annealed at 1273 K for 480 h. Subsequently, all of the samples were cooled down to 673 K with a rate of 10 K/h and kept at 673 K for 240 h, and then quenched in liquid nitrogen.

Samples for X-ray diffraction (XRD) analyses were ground into powders. The XRD data were collected on a Rigaku D/Max 2500V diffractometer with Cu $\rm k_{\alpha}$ radiation and graphite monochromator operated at 40 kV, 200 mA. The Materials Data Inc. software Jade 5.0 [\[29\]](#page-10-0) and Powder Diffraction File (PDF release 2002) [\[28\]](#page-10-0) were used for phase identification. Selected samples were examined by optical microscopy (Leica DMLP, Germany) and then analyzed by scanning electron microscope equipped with energy dispersive X-ray spectroscopy (VEGA3 TESCAN CZECH) in backscattered electron (BSE) imaging modes for microstructure observation and composition measurement.

3. Results and discussion

3.1. Binary compounds

[Table 1](#page-2-0) gives the crystallographic data of the binary and ternary compounds in the Al–Dy–Ge system. Five intermetallic compounds, i.e., $AlDy_2$, Al_2Dy_3 , $AlDy$, Al_2Dy and Al_3Dy presented in the Al–Dy binary phase diagram in the Refs. [\[25,30,31\]](#page-10-0) have been confirmed to exist in our work. Pop et al.[\[32\]](#page-10-0) reported a binary compound $\text{Al}_{17}\text{Dy}_2$ with hexagonal $\text{Ni}_{17}\text{Th}_2$ type structure (space group $P6_{3/mmc}$, a = 1.1788 nm, and c = 1.1322 nm) in the Al–Dy system. In order to identify the existence of the phase $\text{Al}_{17}\text{Dy}_2$, a series of alloy samples with composition in the Al-rich region of the Al–Dy–Ge system were prepared. The calculated XRD pattern of $Al_{17}Dy_2$ was obtained from the crystallographic data in Ref. [\[32\]](#page-10-0) using the LAZY program [\[33\]. B](#page-10-0)y analyzing the XRD patterns of these samples by

Table 1

Crystallographic data of the compounds in the Al–Dy–Ge system at 673 K.

Fig. 2. XRD pattern of the sample No. 8 (Dy_{41.5}Ge_{58.5}) located in the phase region of Dy₂Ge₃ + Dy₃Ge₄ + Dy₂O₃. (There is a minor impure phase of Dy₂O₃, and the peak at 2θ = 29 \circ belongs to the phase of Dy₂O₃.)

using the database of PDF [\[28\]](#page-10-0) and the calculated XRD pattern of $Al₁₇Dy₂$, we were able to identify the phases in each sample. [Fig. 1](#page-1-0) shows the XRD patterns of two selected alloy samples of No. 21 $\rm (Al_{80}Dy_{20})$ and No. 56 $\rm (Al_{55}Dy_{25}Ge_{20})$ indicating the absence of the compound $Al_{17}Dy_2$. From [Fig. 1, i](#page-1-0)t can be seen that the sample No.

21 ($Al_{80}Dy_{20}$) consists of the two phases of Al and Al_3Dy and the sample No. 56 (Al₅₅Dy₂₅Ge₂₀) contained the phases of Al, Al₃Dy (or $\text{Al}_{3-x}\text{DyGe}_x$) and AlDyGe. No evidence was found to confirm the existence of $Al_{17}Dy_2$ under our experimental conditions. This is in good agreement with Ref. [\[25\].](#page-10-0)

Fig. 3. XRD pattern of the sample No. 9 (Dy₄₇Ge₅₃) consisted of the phases of Dy₃Ge₄ and DyGe.

Fig. 4. XRD pattern of the sample No. 72 (Al₅Dy₃₅Ge₆₀) contained the phases of Al_{0.33}DyGe₂, Dy₃Ge₅ and AlDy₂Ge₃.

Seven binary compounds (Dy₅Ge₃, Dy₅Ge₄, DyGe, Dy₂Ge₃, Dy₃Ge₅, DyGe₂ and DyGe₃) in the Dy-Ge system were confirmed to exist at 673 K, which is consistent with the results that pre-sented in Refs. [\[25,34\].](#page-10-0) However, the compound Dy_3Ge_4 in this system reported in Ref. [\[35\]](#page-10-0) was observed under our experimental conditions. The XRD patterns of the samples No. 8 ($Dy_{41.5}Ge_{58.5}$) and No. 9 ($Dy_{47}Ge_{53}$) with composition near Dy_3Ge_4 clearly proves the existence of the compound Dy_3Ge_4 , as shown in [Figs. 2 and 3.](#page-2-0) The samples No. 8 ($Dy_{41.5}Ge_{58.5}$) consists of the phases of Dy_2Ge_3 , Dy_3Ge_4 and impurity phase Dy_2O_3 and No. 9 ($Dy_{47}Ge_{53}$) contained the phases of Dy_3Ge_4 and DyGe. Mokra et al. [\[36\]](#page-10-0) claimed that the compound Dy_6Ge_{11} crystallized in an orthorhombic structure (space group $Cmc2_1$, $a = 0.41027$ nm, $b = 2.9705$ nm, and $c = 0.39316$ nm). By comparing the experimental X-ray diffraction patterns of some alloys with the calculated XRD pattern of the compound Dy_6Ge_{11} obtained from the crystallographic data in Ref.

Fig. 5. XRD pattern of the sample No. 65 ($Al_{15}Dy_{35}Ge_{50}$) located in the phase region of $AlDy_2Ge_3 + AlDyGe + Dy_2Ge_3$.

Fig. 6. XRD pattern of the sample No. 76 (Al₂₀Dy₂₅Ge₅₅) located in the phase region of $Al_{0.33}DyGe_2 + Al_3Dy_2Ge_4 + AlDy_2Ge_6$.

[\[36\],](#page-10-0) the absence of the compound Dy_6Ge_{11} at 673 K was confirmed. The experimental XRD of the sample is more close to that of DyGe₂ [\[27\], t](#page-10-0)herefore, the existence of DyGe₂ is considered at 673 K.

No compound was found in the Al–Ge system.

3.2. Ternary compounds

Four ternary compounds reported in literature, i.e., AlDyGe [\[10\], A](#page-9-0)l₃Dy₂Ge₄ [10], AlDy₂Ge₃ [\[16\], a](#page-10-0)nd AlDy₂Ge₂ [\[24\]](#page-10-0) were confirmed to exist at 673 K. Five new ternary compounds $(Al_{0.33}DyGe₂)$, Al_2DyGe_2 , AlDy₂Ge₆, AlDy₃Ge₃, and Al_{3-x}Dy₁₁Ge_{7+x}) were found in the Al–Dy–Ge system at 673 K. Crystallographic data of all ternary compounds are summarized in [Table 1.](#page-2-0) The ternary compound $Al_{0.33}$ DyGe₂ crystallizes in the orthorhombic CeNiSi₂-type struc-ture [\[37\]](#page-10-0) with space group Cmcm (No. 63) and $a = 0.41018(2)$ nm, $b = 1.62323(6)$ nm, $c = 0.39463(1)$ nm [\[20\]. T](#page-10-0)he compound Al₂DyGe₂ has a La₂O₃-type [\[38\]](#page-10-0) structure with space group $\overline{P3}ml$ (No. 164), and $a = 0.4204$ nm and $c = 0.6681$ nm. The new ternary compound AlDy₂Ge₆ was found to crystallize in the orthorhom-

Fig. 7. XRD pattern of the sample No. 85 ($Al_5Dy_{30}Ge_{65}$) consisted of the three phases of $Al_{0.33}DyGe_2$, $DyGe_2$ and $AlDy_2Ge_6$.

Fig. 8. XRD pattern of the sample No. 46 ($Al_{40}Dy_{36}Ge_{24}$) sit in the phase region of $Al_2Dy + AlDyGe + AlDy_2Ge_2$.

bic Ce₂CuGe₆-type structure with space group $Amm2$ (No. 38) and $a = 0.4144$ nm, $b = 0.3992$ nm and $c = 2.0576$ nm. The new ternary compound $AlDy_3Ge_3$ crystallizes in an orthorhombic structure with space group Pmcn (No. 62) and unit-cell parameters $a = 1.7266(8)$ nm, $b = 0.8175(2)$ nm, and $c = 0.5999(3)$ nm. The analysis of the X-ray powder diffraction patterns revealed that the new ternary compound $Al_{3-x}Dy_{11}Ge_{7+x}$ has a solid solution range of $0 \le x \le 0.7$ at 673 K. The new ternary compound $Al_{3-x}Dy_{11}Ge_{7+x}$ with $x = 0$ crystallizes in a tetragonal structure with space group I4/mmm (No. 139) and unit-cell parameters $a = 1.09816(4)$ nm, and $c = 1.61037(8)$ nm. The unknown crystal structure needs additional study.

The existence of the above ternary compounds can be proved by the X-ray diffraction patterns of the samples located in the threephase regions. Phase identification of several selected ternary alloys using X-ray powder diffraction technique is shown in [Figs. 4–11.](#page-3-0) [Fig. 4](#page-3-0) presents the X-ray diffraction pattern of the sample No. 72 $(Al_5Dy_{35}Ge_{60})$ contained the phases of $Al_{0,33}DyGe_2$, Dy_3Ge_5 and

Fig. 9. XRD pattern (a) and SEM image (b) of the sample No. 40 (Al₂₀Dy₅₂Ge₂₈) located in the three phase region of Al₃Dy₁₁Ge₇ + Dy₅Ge₃ + Al₂Dy.

AlDy₂Ge₃. The sample No. 65 (Al₁₅Dy₃₄Ge₅₀ alloy) consists of the three phases of AlDy₂Ge₃, AlDyGe, and Dy₂Ge₃ seen in [Fig. 5.](#page-4-0) The sample No. 76 $(Al_{20}Dy_{25}Ge_{55}$ alloy) consists of the phases $\text{Al}_{0.33}\text{DyGe}_2$, $\text{Al}_3\text{Dy}_2\text{Ge}_4$, and AlDy_2Ge_6 seen in [Fig. 6.](#page-4-0) The XRD analysis of the samples $Al₅Dy₃₀Ge₆₅$ shows the existence of the three-phase region of $\text{Al}_{0,33}\text{DyGe}_2$ + DyGe_2 + AlDy_2Ge_6 as shown in [Fig. 7. T](#page-5-0)he XRD pattern of the sample No. $46 (Al_{40}Dy_{36}Ge_{24})$ located in the phase region of $Al_2Dy + AlDyGe + AlDy₂Ge₂$ is given in [Fig. 8.](#page-5-0) Fig. 9 presents the X-ray diffraction pattern (a) and the SEM image (b) of the sample No. 40 $(Al_{20}Dy_{52}Ge_{28})$ contained of the three phases of $Al_{3-x}Dy_{11}Ge_{7+x}$, Dy₅Ge₃, and Al₂Dy. [Fig. 10](#page-7-0) shows the X-ray diffraction pattern (a) and the SEM image (b) of the sample No. 42 ($Al_{20}Dy_{45}Ge_{35}$). The X-ray diffraction analysis for the sample gives the same result with that of SEM analysis. It can be clearly seen from [Fig. 10](#page-7-0) that the sample consisted of the three phases of $Al_{3-x}Dy_{11}Ge_{7+x}$, $AlDy_2Ge_2$ and Al_2Dy . The X-ray diffraction pattern of the sample No. 52 $(AlgDy_{44}Ge_{48})$ is shown in [Fig. 11](#page-8-0) proving the existence of the compound $AlDy_3Ge_3.$

3.3. Pseudo-binary compound

A series samples with compositions of $\text{Al}_{3-x}\text{DyGe}_x$ with $x = 0.1$, 0.2, 0.3, 0.4, 0.5 were prepared by arc-melting. The XRD patterns of the series samples of $\text{Al}_{3-x}\text{DyGe}_x$ are shown in [Fig. 12. T](#page-8-0)he XRD analysis pointed out that the obtained samples $\text{Al}_{3-x}\text{DyGe}_x$ with

Fig. 10. XRD pattern (a) and SEM image (b) of the sample No. 42 (Al₂₀Dy₄₅Ge₃₅) in the three phase region of Al₃Dy₁₁Ge₇ + AlDy₂Ge₂ + Al₂Dy.

 $x \le 0.3$ contained the single phase of Al_{3−x}DyGe_x. It also can be seen from [Fig. 12,](#page-8-0) the samples Al_{3-x} DyGe_x with x > 0.3 consisted of a small amount second and third phases of $Al₂DyGe₂$ and AlDyGe. Therefore, the pseudo-binary compound $\text{Al}_{3-x}\text{DyGe}_x$ existed in the range of $x \le 0.3$ (with maximum of 7.5 at.% Ge), which are in agreement with Ref. [\[17\].](#page-10-0)

3.4. Isothermal section at 673 K

The isothermal section of the Al–Dy–Ge system at 673 K has been determined by comparing and analyzing the X-ray diffraction patterns of 92 binary and ternary alloys combined with the aid of the scanning electron microscopy in the present work. The isothermal section consists of 25 single-phase regions, 56 two-phase regions and 32 three-phase regions, as shown in [Fig. 13.](#page-8-0) There are nine ternary compounds AlDyGe, $\text{Al}_3\text{Dy}_2\text{Ge}_4$, Al Dy_2Ge_3 , Al Dy_2Ge_2 , Al $_{0.33}\text{Dy}\text{Ge}_2$, Al Dy_2Ge_6 , Al $_2\text{Dy}\text{Ge}_2$, AlDy₃Ge₃, Al_{3-x}Dy₁₁Ge_{7+x} ($x \le 0.7$) and one pseudo-binary compound Al_{3-x} DyGe_x existing in the system. The nominal composition and phase components of each sample in the isothermal section of the Al–Dy–Ge ternary system at 673 K are given in [Table 2.](#page-9-0)

Fig. 11. XRD pattern of the sample No. 52 (Al₈Dy₄₄Ge₄₈) contained the three phases of Dy₃Ge₄, DyGe and AlDy₃Ge₃.

Fig. 12. XRD patterns of the compound Al_{3−x}DyGe_x.

Fig. 13. Isothermal section of the Al–Dy–Ge system at 673 K.

Table 2

The nominal composition and phase components of each sample in the isothermal section of the Al–Dy–Ge ternary system at 673 K.

4. Conclusions

The isothermal section of the Al–Dy–Ge system at 673 K has been determined by comparing and analyzing the X-ray diffraction patterns of 92 binary and ternary alloys combined with the aid of the scanning electron microscopy in the present work. The isothermal section consists of 25 single-phase regions, 56 two-phase regions and 32 three-phase regions, as shown in [Fig. 13.](#page-8-0) There are nine ternary compounds AlDyGe, $\mathrm{Al}_{3}\mathrm{Dy_{2}Ge_{4}}$
 AlDy₂Ge₃, AlDy₂Ge₂, Al_{0.33}DyGe₂, AlDy₂Ge₆, Al₂DyGe₂, AlDy₃Ge₃, Al_{3-x}Dy₁₁Ge_{7+x} ($x \le 0.7$) and one pseudo-binary compound Al_{3-x} DyGe_x existing in the system. The nominal composition and phase components of each sample in the isothermal section of the Al–Dy–Ge ternary system at 673 K are given in Table 2.

Acknowledgement

This work was supported by the National Natural Science Foundation of China (No. 50861005 and 50961002).

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