Phase equilibria of the Cu–Dy–Ti ternary system at 973 K

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The solid-state phase equilibria of the copper (Cu)–dysprosium (Dy)–titanium (Ti) ternary system at 973 K has been experimentally investigated. The existence of nine binary compounds, Cu_4Ti, Cu_3Ti_2, Cu_4Ti_3, CuTi, $CuTi_2$, $CuTi_3$, CuDy, Cu_2Dy, and Cu_5Dy was confirmed. The controversial phase of $CuTi_3$ was found in this work. The temperature range of Cu_7Dy was determined to be from 1112 to 1183 K. The phase relations at 973 K are governed by ten ternary phase regions, 21 binary phase regions, and 12 single-phase regions. The solid solubility of Cu in Dy is undetectable. None of the other phase in this system reveals a remarkable homogeneity range at 973 K. © *2015 International Centre for Diffraction Data.* [doi:10.1017/S0885715615000287]

Key words: Cu-based alloy, Cu–Dy–Ti system, phase equilibrium

I. INTRODUCTION

Owing to the excellent combination of high electrical conductivity and strength, copper (Cu)–beryllium (Be) alloys are used for numerous applications such as lead frames, electrical connectors, elastic components, and precise instruments (Wilkes, 1968). To avoid environmental pollution because of the toxic character of Be, advanced metallic materials are required to be developed to replace Cu–Be alloys. Therefore, worldwide research has been aimed at developing a substitute for the noxious and costly Cu–Be alloys. Age-hardenable copper–titanium (Cu–Ti) alloys containing approximately 1–6 at.% Ti are attractive as a candidate since their mechanical strength is comparable with Cu–Be alloys with higher wear resistance and superior stress relaxation behavior (Datta and Soffa, 1976; Nagarjuna *et al.*, 1999; Soffa and Laughlin, 2004). Unfortunately, their electrical conductivity is rather low (Nagarjuna *et al.*, 1997; Suzuki *et al.*, 2003).

To obtain a good balance between conductivity and strength, much work has been done (Nagarjuna *et al.*, 1999; Kamegawa *et al.*, 2010, 2013; Semboshi, 2007; Semboshi *et al.*, 2009, 2011a, 2011b). Semboshi *et al.* (2011a, 2011b) made a systematic investigation of the Cu–Ti alloys and found that the electrical conductivity and the strength of Cu–(1–6) at.% Ti alloy increased greatly when aging in a hydrogen (H_2) atmosphere. Moreover, they demonstrated that good balance between conductivity and strength can be obtained by aging at low temperature and high H_2 pressure and by prior deformation of cold rolling (Semboshi *et al.*, 2011a, 2011b). Recently, high-strength and high-conductivity Cu–4.2 mol% Ti alloy wire was fabricated by Semboshi and Takasugi (2013).

Mechanical properties of Cu–Ti alloys are limited to intermediate temperatures up to 723 K. Unfortunately, these properties drop down at higher temperatures because of the coarsening of the β-phase precipitates with the nominal composition of Cu_4Ti (Kato *et al.*, 1980; Ardell, 1985). As an important kind of alloying additive for metallic materials, rare earth (RE) can significantly improve the properties of alloys by affecting the microstructure and refining grain. To discover further application characteristics and regularities concerning phase formation in the Cu–Ti–RE ternary system, it is necessary to investigate phase relationships in this system. Up to now, reports on the Cu–Ti–RE phase diagrams are limited, except the Cu–Ti–Y (Hu *et al.*, 2009) and Cu–Ti–Er ternary systems (Zhan *et al.*, 2012). The work presented in this paper is aiming to determine the Cu–dysprosium (Dy)–Ti phase equilibria at 973 K.

II. BINARY SYSTEMS

A. Cu–Ti binary system

For the Cu–Ti binary system, the existence of six intermediate phases, i.e., $CuTi_2$, CuTi, Cu_4Ti_3, Cu_3Ti_2, Cu_4Ti, and Cu_2Ti is accepted without question. But the existence of the phase $CuTi_3$ and the stability range of the phase Cu_2Ti is controversial. Eremenko *et al.* (1966) reported that the temperature for the invariant reaction $Cu_2Ti \leftrightarrow Cu_4Ti + Cu_3Ti_2$ was 1123 K; however, this temperature suggested by Murray (1983) was about 1143 K. Canale and Servant (2002) suggested that $CuTi_3$ was a stable phase on the basis of differential thermal analysis results. Kumar *et al.* (1996) also reported the same results when thermodynamically evaluated the Cu–Ti system. After that, Karlsson (1951) reported the possible existence of $CuTi_3$. Recently, Zhan *et al.* (2012) confirmed the $CuTi_3$ in the microstructure of the alloy 25Cu75Ti. They heated the 25Cu75Ti alloys to 1023 K and then kept warm for 90 h. The temperature of the eutectoid transformation, namely, $\beta Ti \leftrightarrow \alpha Ti + CuTi_3$, was determined to be 1078 K in their work. They suggested that the formation of the $CuTi_3$ phase was controlled by the annealing temperature and annealing time. At the same time, it should be a metastable phase in light of the works carried out by the other groups (Massalski *et al.*, 1986; Xu *et al.*, 2005; Liu *et al.*, 2006a; Wang *et al.*, 2006)

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B. Cu–Dy binary system

For the Cu–Dy binary system, much work has been done so far (Baenziger and Moriarty, 1961; Storm and Benson, 1963; Copeland and Kato, 1964; Buschow *et al.*, 1969; Buschow and Van Der Goot, 1971; Zheng and Xu, 1982; Franceschi, 1982; Subramanian and Laughlin, 1988; Zhang *et al.*, 2009). The first systematical study of the Cu–Dy system was performed experimentally by Zheng and Xu (1982). At the same time, the Cu–Dy system was also investigated experimentally by Franceschi (1982). Zheng and Xu (1982) reported that the maximum solid solubility of Cu in Dy was 12.5 at.% Cu, while Franceschi (1982) reported that there was no appreciable solid solutions of Cu in Dy. Subramanian and Laughlin (1988) assessed the Cu–Dy system and most of their work was accepted except the stable temperature range of Cu_7Dy phase and the solid solubility of Cu in Dy. Zhang *et al.* (2009) thought that the result of Zheng and Xu (1982) was better than that of Franceschi (1982) because the properties of Gd and Dy were quite similar and the solid solubility of Cu in Gd was 15 at.% (Carnasciali *et al.* 1983a). To obtain a convincible result, Zhang *et al.* (2009) reassessed the Cu–Dy system and found that the maximum terminal solubility of Cu in Dy was 12 at.% Cu.

CuDy, Cu_2Dy, and Cu_5Dy have been confirmed so far. About the Cu_5Dy, Buschow *et al.* (1969) found Cu_5Dy_H and Cu_5Dy_L phases. For the Cu_7Dy, much investigation confirmed that it was a high-temperature phase, but the range of the temperature was ambiguous. Buschow and Goot (1971) found that the Cu_7Dy was not stable below 973 K, while Zheng and Xu (1982) found that the temperature range of existence of Cu_7Dy was from 1121 to 1163 K. However, later in Franceschi' s work (1982), this compound was reported to be stable between 1048 and 1133 K. So to obtain a more reliable result, Zhang *et al.* (2009) reassessed the Cu–Dy system and they found that the temperature range of existence of Cu_7Dy was from 1054 to 1174 K.

To resolve the differences, the solid solubility of Cu in Dy and the range of the temperature of Cu_7Dy are restudied in this work, which will be discussed in the phase analysis part.

C. Dy–Ti binary system

The previous work has indicated that no binary compound exists in the Dy–Ti system (Zhuang *et al.*, 1996; Bulanova *et al.*, 2004; Liu *et al.*, 2006b; Yan *et al.*, 2009). Linus Pauling file entry prototype of the binary Cu–Ti and Cu–Dy phases are summarized in Table I. Additionally, the lattice parameters calculated in this work are also showed in Table I.

III. EXPERIMENTAL DETAILS

The alloy samples were produced by arc melting on a water-cooled Cu cast with a non-consumable tungsten electrode under pure argon atmosphere. Titanium was used as O_2 getter during the melting process. The sample was prepared with a total weight of 2 g and the purity of Ti, Cu, and Dy were all 99.9 wt.%. The arc-cast button was turned around after each melting and remelted at least three times for better homogeneity. In total, 75 samples were prepared and the weight losses were almost all <1% after melting. The melted alloy buttons were sealed in small glass tubes for homogenization heat treatment. The homogenization temperatures of the samples were determined according to the phase diagrams of the

TABLE I. Binary crystal structures in the Cu–Dy–Ti system quenched from 973 K.

Compound	The Linus Pauling file entry structure type	Space group	Pearson symbol	Lattice parameters (nm) Measured at room temperature			Reference
				a	*b*	*c*	
$CuTi_3$	Cu_3Ti	*Pbam*	oP8	1.203 57	1.198 02	0.298 72	Canale and Servant (2002)
				0.4158	—	0.3594	Karlsson (1951)
				0.413 13	—	0.358 76	This work
$CuTi_2$	$CuZr_2$	*I4/mmm*	tI6	0.294 38	—	1.0786	Villars (1997)
				0.295 99	—	1.0782	This work
CuTi	CuTi	*P4/nmm*	tP4	0.3108	—	0.5887	Villars (1997)
				0.4430	—	0.2843	This work
Cu_4Ti_3	Cu_4Ti_3	*I4/mmm*	tI14	0.312	—	1.994	Villars (1997)
				0.3121	—	1.990	This work
Cu_3Ti_2	Cu_3Ti_2	*P4/nmm*	oS12	0.313	—	1.395	Villars (1997)
				0.3143	—	1.398	This work
Cu_2Ti	Au_2V	*Amm2*	oC12	0.438	0.797	0.449	Schubert (1965)
βCu_4Ti	Au_4Zr	*Pnma*	oP20	0.4522	0.4344	1.2897	Villars (1997)
				0.4508	0.4338	1.294	This work
CuDy	ClCs	$Pm\bar{3}m$	cP2	0.3462	—	—	Morrin and Pierre (1974)
				0.3461	—	—	Zheng and Xu (1982)
				0.3455	—	—	Franceschi (1982)
				0.3474	—	—	This work
Cu_2Dy	$CeCu_2$	*Imma*	oI12	0.4300	0.6792	0.7300	Copeland and Kato (1964)
				0.4287	0.6852	0.7285	This work
Cu_5Dy_L	$AuBe_5$	$F\bar{4}3m$	cF24	0.7027	—	—	Buschow *et al.* (1969)
				0.7047	—	—	Zheng and Xu (1982)
				0.7022	—	—	Franceschi (1982)
				0.7028	—	—	This work
Cu_5Dy_H	$CaCu_5$	*P6/mmm*	*hP6*	0.502	—	0.408	Carnasciali *et al.* (1983b)

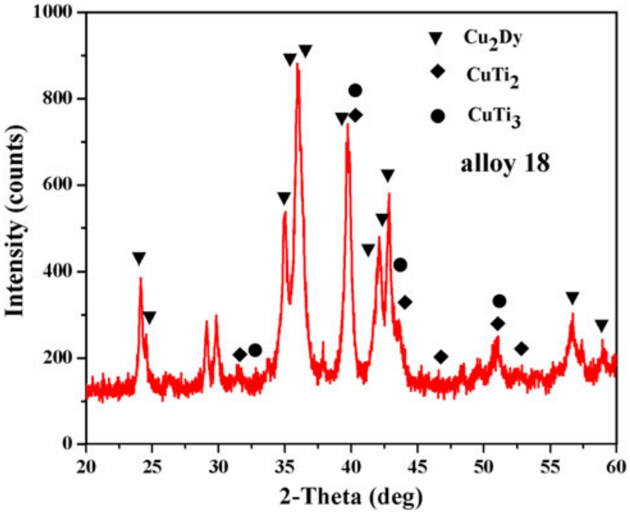

Figure 1. (Color online) The XRD pattern of #18 sample (44 at.% Cu, 18 at.% Dy, and 38 at.% Ti) indicating the existence of CuTi$_3$, CuTi$_2$, and Cu$_2$Dy.

Cu–Ti, Dy–Ti, and Cu–Dy systems and the results of differential scanning calorimetry (DSC) of some typical ternary alloys. The alloys were annealed at 973 K for more than 1 month. Finally, the samples were quenched in ice-water.

Room temperature X-ray powder diffraction (XRD) and scanning electron microscopy (SEM) with energy dispersive analysis (EDX) were used in the present investigation. Samples for the XRD analysis were initially placed in a metal mortar and the pestle was used to break apart the sample and then repeated grinding, until it becomes powder (the finer, the better). Usually, the mean particle size should be 30–50 μm. The samples were measured with the help of Rigaku D/Max 2500 V diffractometer with Cu$K\alpha$ radiation and graphite monochromator operated at 40 kV and 200 mA. The scan ranges of the samples were from 20° to 60° (2θ) with a scanning speed of 10° min^{-1}. The Materials Data Inc. software Jade 5.0, PCW (powder Cell Windows) software and PDF-2 2010 Inorganic (Dr. Soorya Kabekkodu, 2010) were used for phase identification. For the SEM/EDX analyses, the alloys were prepared by following the standard metallographic procedures: hot mounting in resin, grinding in the sequence of no. 400–3000 SiC sand paper, and polishing with colloidal silica suspension (OP-S). DSC experiments were performed by DSC. The DSC curve (heat flow vs. temperature) of alloy Cu$_7$Dy was recorded at a rate of 10 K min^{-1} under a flow of pure argon on the NETZSCH STA 449 C instrument with the crucible-type DSC/TG. Pan A1$_2$O$_3$ and TAB separator are used, and the temperature was calibrated by pure aluminum. By all these means, the phase relations of the Cu–Dy–Ti ternary system were determined.

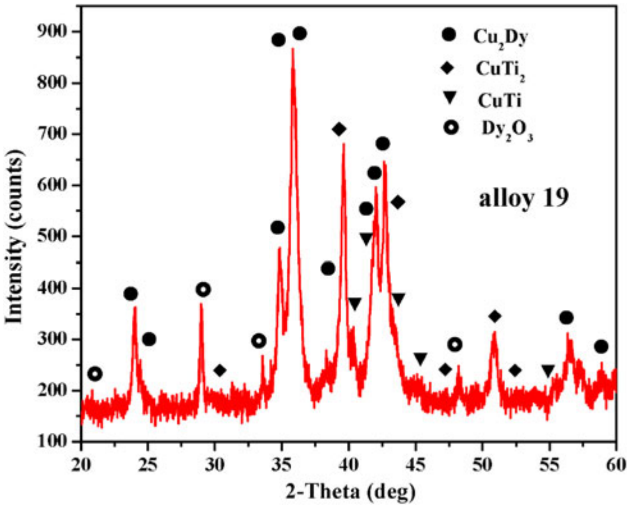

Figure 2. (Color online) The XRD pattern of #19 sample (57 at.% Cu, 20 at.% Dy, and 23 at.% Ti) indicating the existence of CuTi, CuTi$_2$, and Cu$_2$Dy.

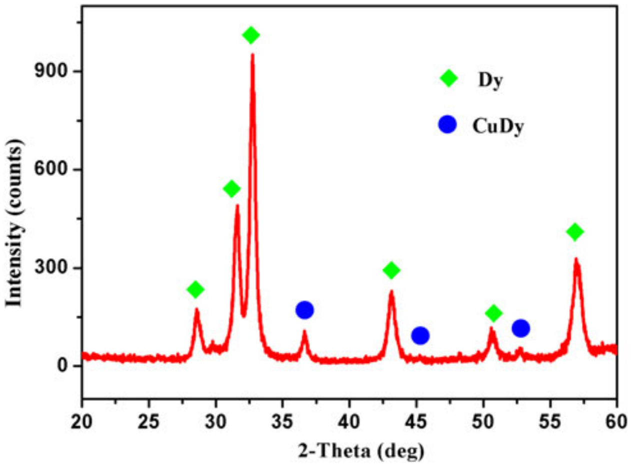

Figure 3. (Color online) The XRD pattern of the equilibrated alloy of the Cu$_1$Dy$_{99}$.

IV. RESULT AND DISCUSSION

A. Phase analysis

In the Cu–Ti system, the existence of six compounds, i.e., CuTi$_3$, CuTi$_2$, CuTi, Cu$_4$Ti$_3$, Cu$_3$Ti$_2$, and Cu$_4$Ti has been confirmed at 973 K. Canale and Servant (2002) suggested that the phase CuTi$_3$ was a stable phase on the basis of their experimental results, though it was generally regarded as a metastable phase. Fortunately, in our work, CuTi$_3$ was observed from the XRD patterns of the equilibrated alloys with composition equal to or near to Cu:Ti = 1: 3. Furthermore, the XRD pattern of #18 sample (44 at.% Cu, 18 at.% Dy, and 38 at.% Ti) clearly indicates the existence of CuTi$_3$ (25-1144), CuTi$_2$ (72-0441),

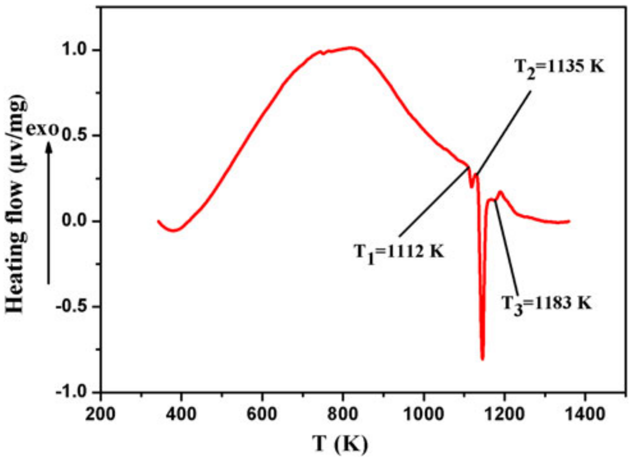

Figure 4. (Color online) DSC heating curve of the alloy Cu$_7$Dy.

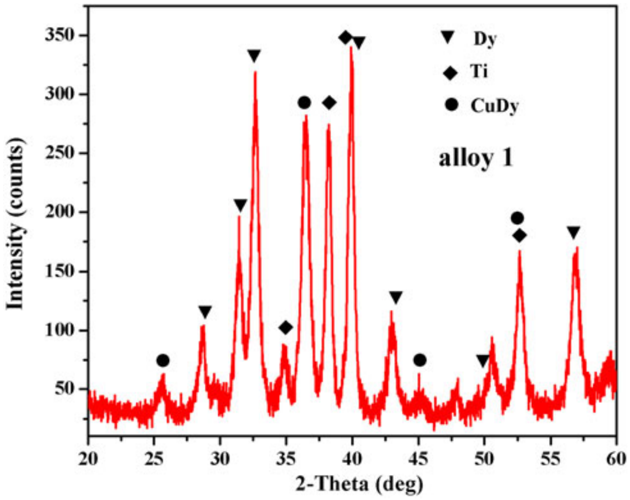

Figure 5. (Color online) The XRD pattern of #1 sample (25 at.% Cu, 50 at.% Dy, and 25 at.% Ti) containing Ti, Dy, and CuDy.

and Cu_2Dy (39-1344), as shown in Figure 1, which is in accordance with the previous reports from Zhan *et al.* (2012) and Eremenko *et al.* (1966). The XRD pattern of #19 sample (57 at.% Cu, 20 at.% Dy, and 23 at.% Ti) shows the existence of CuTi (07-0114), $CuTi_2$ (72-0441), and Cu_2Dy (39-1344), as shown in Figure 2.

In the Cu–Dy binary system, CuDy, Cu_2Dy, and Cu_5Dy have been confirmed at 973 K, which can be seen from the XRD pattern or the SEM micrograph in Figures 1 and 2 mentioned above and in the following Figures 5, 6 and 8.

Figure 3 shows the XRD pattern of the equilibrated alloy of the Cu_1Dy_{99}. It is obviously seen that CuDy can be detected, which reveals that Cu does not show remarkable solubility in Dy, which is in agreement with the result from Franceschi (1982).

The DSC curve (heat flow vs. temperature) of alloy Cu_7Dy is shown in Figure 4. The change in the slope of the heat flow temperature curve can be observed. The temperature of the extrapolated onset is used as the phase transformation temperature in this work, so T_1, T_2, and T_3 represent three phase transformation temperatures, respectively. An obvious endothermic peak related to the eutectic transformation $L \Leftrightarrow Cu + Cu_7Dy$ is observed at $T_2 = 1135$ K. The temperature T_1 is related to the phase transition temperature of the eutectoid reaction, namely, $Cu_7Dy \Leftrightarrow Cu + Cu_5Dy\_L$ at 1112 K. The temperature T_3 is related to the peritectic transition action $L + Cu_5Dy\_L \Leftrightarrow Cu_7Dy$. The temperature range of the existence of Cu_7Dy is from 1112 to 1183 K, which agrees well with the work of Zheng and Xu (1982). Table II shows the reactions in this work and the results from previous references (Zheng *et al.*, 1982; Franceschi, 1982; Subramanian *et al.*, 1988).

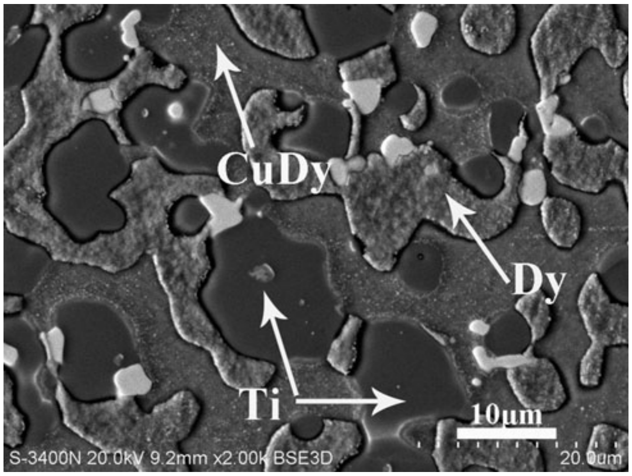

Figure 6. SEM micrograph of the equilibrated alloy 25 at.% Cu, 50 at.% Dy, 25 at.% Ti containing Ti, Dy, and CuDy. (The white areas relate to the Dysprosium oxide.)

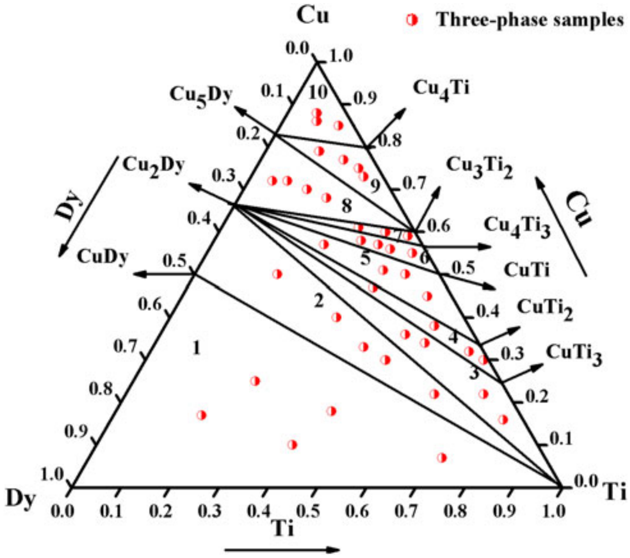

Figure 7. (Color online) The phase diagram (with the present experimental data) of the Cu–Dy–Ti ternary system at 973 K.

Figure 5 shows the XRD pattern of #1 sample (25 at.% Cu, 50 at.% Dy, and 25 at.% Ti), illustrating the existence of Ti (44-1294), Dy (89-2926), and CuDy (65-4143). The SEM photograph in Figure 6 also clearly indicates the existence of the above phases (identified by EDX). In the Dy–Ti system, it is confirmed that no binary compound exists in this work, which is in good agreement with the results from (Zhuang *et al.*, 1996; Bulanova *et al.*, 2004; Liu *et al.*, 2006b; Yan *et al.*, 2009).

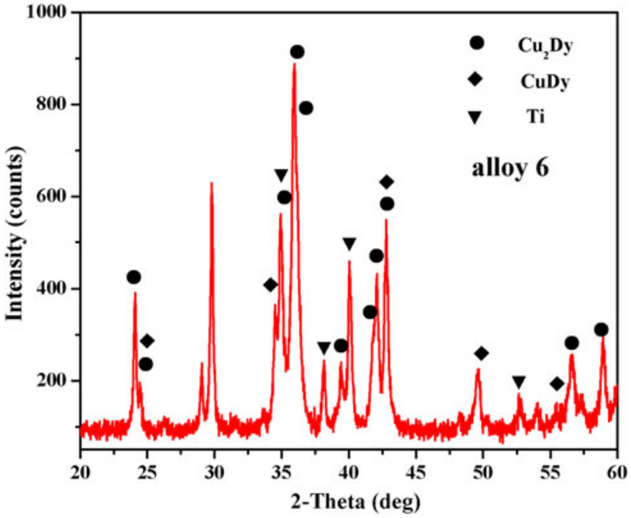

Figure 8. (Color online) The XRD pattern of #6 sample (40 at.% Cu, 26 at.% Dy, and 34 at.% Ti) illustrates the existence of Ti, CuDy, and Cu_2Dy.

TABLE II. Invariant reactions of the Cu_7Dy in the Cu–Dy system.

Reaction	T (K)	Reaction type	Reference
$L \Leftrightarrow Cu + Cu_7Dy$	1153	Eutectic	Zheng and Xu (1982)
	1157	Eutectic	Zhang *et al.* (2009)
	1135	Eutectic	This work
$L + Cu_5Dy\_L \Leftrightarrow Cu_7Dy$	1165	Peritectic	Zheng and Xu (1982)
	1133	Peritectic	Franceschi (1982)
	1174	Peritectic	Zhang *et al.* (2009)
	1183	Peritectic	This work
$Cu_7Dy \Leftrightarrow Cu + Cu_5Dy\_L$	1121	Eutectoid	Zheng and Xu (1982)
	1048	Eutectoid	Franceschi (1982)
	1054	Eutectoid	Zhang *et al.* (2009)
	1112	Eutectoid	This work

In the previous work, no ternary compound has been reported in the ternary Cu–Dy–Ti system. This result is also confirmed in the present work (at 973 K).

TABLE III. Details of the phase regions and typical samples in the Cu–Dy–Ti system at 973 K.

Phase regions	Specimens	Phase composition (at.%)			Phases (ICDD-PDF number)
		Cu	Dy	Ti	
1	1	25	50	25	Ti (44-1294)
	2	17	65	18	Dy (89-2926)
	3	7	21	72	CuDy (65-4143)
	4	10	50	40	
	5	18	38	44	
2	6	40	26	34	Ti (44-1294)
	7	33	24	43	CuDy (65-4143)
	8	50	33	17	Cu_2Dy (39-1344)
	9	30	21	49	
	10	22	15	63	
3	11	36	14	50	Ti (44-1294)
	12	16	4	80	$CuTi_3$ (25-1144)
	13	22	5	73	Cu_2Dy (39-1344)
	14	34	11	55	
4	15	30	1	69	$CuTi_3$ (25-1144)
	16	32	3	65	$CuTi_2$ (72-0441)
	17	38	7	55	Cu_2Dy (39-1344)
	18	44	18	38	
5	19	57	20	23	CuTi (07-0114)
	20	50	7	43	$CuTi_2$ (72-0441)
	21	51	11	38	Cu_2Dy (39-1344)
	22	45	5	50	
6	23	56	7	37	CuTi (07-0114)
	24	57	9	34	Cu_4Ti_3 (18-0460)
	25	55	3	42	Cu_2Dy (39-1344)
	26	58	12	30	
7	27	61	11	28	Cu_3Ti_2 (18-0459)
	28	60	6	34	Cu_4Ti_3 (18-0460)
	29	59	2	39	Cu_2Dy (39-1344)
8	30	72	20	8	Cu_3Ti_2 (18-0459)
	31	70	17	13	Cu_2Dy (39-1344)
	32	72	23	5	Cu_5Dy_L (47-0995)
	33	68	14	18	
9	34	79	10	11	Cu_3Ti_2 (18-0459)
	35	73	4	23	Cu_4Ti (20-0370)
	36	77	6	17	Cu_5Dy_L (47-0995)
	37	75	4	21	
10	38	86	7	7	Cu (70-3038)
	39	88	6	6	Cu_4Ti (20-0370)
	40	85	3	12	Cu_5Dy_L (47-0995)

B. Isothermal section

The isothermal section of the Cu–Dy–Ti ternary system at 973 K has been determined on the basis of XRD and SEM, as shown in Figure 7. This isothermal section consists of ten ternary phase regions, 21 binary phase regions, and 12 single-phase regions. Two ternary phase regions, i.e., $CuTi_2 + CuTi_3 + Cu_2Dy$ and $CuTi_3 + Ti + Cu_2Dy$, are supposed based on the stability of the $CuTi_3$ phase. The XRD pattern of #6 sample (40 at.% Cu, 26 at.% Dy, and 34 at.% Ti) illustrates the existence of Ti (44-1294), CuDy (65-4143), and Cu_2Dy (39-1344), as shown in Figure 8.

The constitutions of the ternary phase regions and compositions of the typical alloys are listed in Table III. The XRD results confirm that nine binary compounds, namely Cu_4Ti, Cu_3Ti_2, Cu_4Ti_3, CuTi, $CuTi_2$, $CuTi_3$, CuDy, Cu_2Dy, and Cu_5Dy exist in this system at 973 K.

The solid solubility ranges in the isothermal sections are determined using the phase-disappearing method and comparing the XRD patterns of samples near the compositions of the binary phases. Variation of the lattice parameters was determined to obtain the solid solubility. None of the other phase found in this system has a remarkable homogeneity range at 973 K.

V. CONCLUSIONS

In this work, the nine binary compounds, i.e., Cu_4Ti, Cu_3Ti_2, Cu_4Ti_3, CuTi, $CuTi_2$, $CuTi_3$, CuDy, Cu_2Dy, and Cu_5Dy have been confirmed. No binary compound is found in the Dy–Ti binary system at 973 K. The isothermal section of the Cu–Dy–Ti ternary system at 973 K consists of 12 single-phase regions, 21 binary phase regions, and ten ternary phase regions. The temperature range of Cu_7Dy is from 1112 to 1183 K. The solid solubility of Cu in Dy is undetectable. None of the other phase in this system shows a remarkable solid solution at 973 K. No ternary compound is found in the present work.

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