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Interdiffusion behaviors and mechanical properties of Cu-Zr system

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ABSTRACT

In this work, solid-to-solid diffusion couples were assembled and annealed to investigate diffusion behavior and mechanical properties of the Cu-Zr system in temperatures range from 1043 K to 1113 K. Six intermetallic compounds (IMCs) Cu_9Zr_2, $Cu_{51}Zr_{14}$, Cu_8Zr_3, $Cu_{10}Zr_7$, CuZr, and $CuZr_2$ were observed in the diffusion zone. Composition-dependent interdiffusion coefficients of IMCs have been calculated based on the measured composition Cu profiles of the diffusion zones by using Sauer–Freise method. And the average effective interdiffusion coefficients for each phase were also calculated by using Wagner method. The activation energies of diffusion are evaluated according to the average effective interdiffusion coefficient. Finally, the load-displacement curves measured by nano-indentation are obtained to characterize mechanical properties of Cu_9Zr_2 and $Cu_{51}Zr_{14}$, which have similar hardness and elastic moduli.

1. Introduction

Diffusion is a common phenomenon in processing of a material, such as carburizing technology, doping in semiconductors, welding in metal materials and aging treatment. Many processes of the material manufactures and performance, such as homogenization of casting components, creep of metals in high temperature, corrosion, oxidation and phase transformation etc. are all related to diffusion. Therefore , the investigation of diffusion has great significance in theory and practice. Diffusion couple technology is a very valuable and high efficiency in studying phase equilibrium and diffusion phenomenon.

Cu-Zr alloys have been extensively studied as a typical binary amorphous system due to its strong amorphous forming ability and easy forming bulk metallic glasses [1–8]. As we known, the glass forming ability of binary or multiple Cu-Zr and/or Cu-Zr-based amorphous alloys made by solid state amorphization is related with both of the thermodynamic and dynamic properties (eg. diffusion coefficients) [9–15]. The stability of equilibrium intermetallic compounds of Cu-Zr and their mechanical properties have been studied extensively [16] due to their outstanding properties (such as ultrahigh-yield strength, large elastic strain limits, high hardness, corrosion resistance and low fracture toughness) and extensive application in structural, chemical and magnetic fields [6–8]. In addition, the development of Cu-Zr amorphous alloy needs both thermodynamic and kinetic knowledge. Although many thermodynamic studies including thermodynamic modeling [13–15] and phase diagram [17–23] have been recently performed. Using CALPHAD method, the binary Cu-Zr phase diagram (Fig. 1) has been assessed [20,23], and the Cu_5Zr_8 is a stable phase [20,23]. However, the recently experiment shows that there are eight compounds, namely Cu_5Zr, $Cu_{51}Zr_{14}$, Cu_8Zr_3, Cu_2Zr, $Cu_{24}Zr_{13}$, $Cu_{10}Zr_7$, CuZr and $CuZr_2$, but the Cu_5Zr_8 phase has not observed [22]. It should be noted that the Cu_5Zr phase is usually denoted as Cu_9Zr_2 [20,21,23,24]. The reaction diffusion between the pure Cu and Zr has been investigated with temperature range from 930 K to 1153 K, the growth kinetics of intermetallic compound layers have been studied and the interdiffusion coefficients of the Cu_9Zr_2 and $Cu_{51}Zr_{14}$ have been calculated [24]. However, the interdiffusion coefficients of the other compounds have not been reported anywhere, therefore, the motivation of this work is to obtain the more diffusion information of Cu-Zr system by using diffusion couple technique.

In this work, interdiffusion between Zr and Cu was examined by using solid-to-solid diffusion couples. The hardness and Young's modulus of the IMCs were measured by nanoindentation. The remainder of this paper is organized as follows. In Section 2, the experimental procedure is described. In Section 3, the diffusion behaviors and mechanical properties are presented and discussed. Finally, brief conclusions are given in Section 4.

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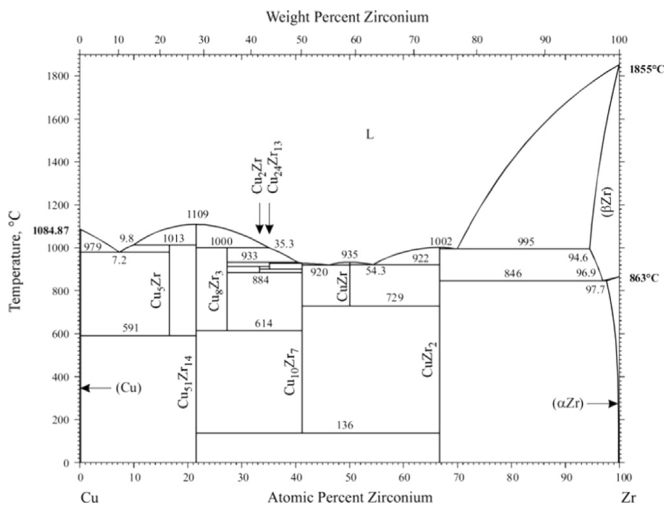

Fig. 1. Phase diagram of the Cu-Zr system.

2. Experimental procedure

Zr (99.9 wt%) and Cu (99.99 wt%) were sectioned into bars with 6 mm in length and 3 mm in thickness. The bars were annealed at 723 K (450 °C for 12 h) in order to relieve any stress during the sample section and obtain a large grain size. Small diffusion disks specimens were mechanically polished on # 240–3000 SiC water matte paper, and then finished using diamond with diameter of 1 µm to obtain mirror-like quality surface. Disks of Zr and Cu were tied with stainless steel jig to insure the diffusion surface of Zr and Cu contacted with each other perfectly. Then the diffusion couples were placed into quartz capsule which was evacuated in order to avoid oxidation and backfilled with ultrahigh purity argon to reach 10^4 Pa inside during the diffusion annealing. Diffusion couple experiments were carried out in a tube furnace at 1043 K , 1073 K, 1093 K and 1113 K for 504, 336, 240 and 168 h, respectively. After the annealing, the capsule was taken from furnace and quenched into ice water to keep high-temperature microstructures. Each diffusion couples were examined firstly under optics microscopy to observe whether the diffusion layers formed.

Electron probe microanalysis (EPMA, JEOL-JXA 8230, Tokyo, Japan) wavelength dispersive spectroscopy (WDS) was employed to determine the concentration profiles of Zr and Cu for each diffusion couple using quantitative analysis of two points scanning modes with a 0.1 µm step in Cu_8Zr_3, CuZr, $CuZr_2$ phases and a 1.0 µm step in Cu_9Zr_2, and $Cu_{51}Zr_{14}$ phases. The accelerating voltage is 15 kV and beam current is 20 nA. More than 10 random positions were taken to determine the layer thickness of each intermetallic from backscatter image (BEI). A ZAF correction was employed for converting the X-ray intensity to the concentration. Nanoindentation was performed with a Nanovea M1 hardness tester using a diamond Berkovich tip. Fifteen indentations were made on each phase. The indentation loading profile consisted of 60 s loading to a peak load, followed by 3 s hold segment, and then 60 s unloading, and the peak load was 20 mN.

3. Results and discussion

3.1. Microstructure of diffusion zone

Fig. 2(a) demonstrates the back scattered image of the diffusion couple sample annealed at 1113 K for 168 h, and the corresponding concentration profile determined by EPMA is presented in Fig. 2(b). According to the binary Cu-Zr phase diagram, eight IMCs exist [22]. However, in the present work, six compounds (Cu_9Zr_2, $Cu_{51}Zr_{14}$, Cu_8Zr_3, $Cu_{10}Zr_7$, CuZr and $CuZr_2$) have been experimentally observed in all diffusion couples in the temperature range of 1043–1113 K based

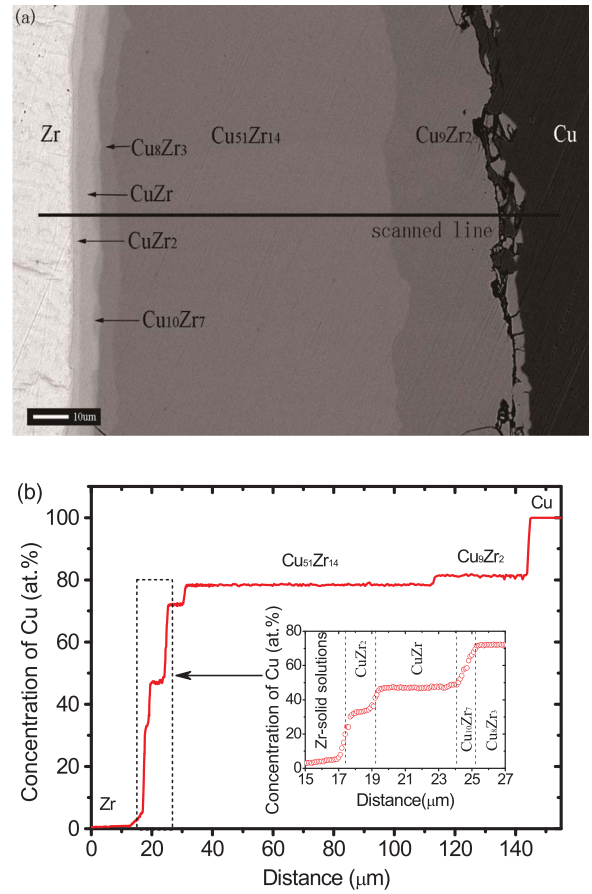

Fig. 2. (a) Back scattered electron micrograph in the interdiffusion zone, and (b) concentration profiles of Zr and Cu determined by electron probe microanalysis from Zr-Cu diffusion couples annealed at for 1113 K for 168 h.

on electron probe microanalysis (EPMA). The present results are in good agreement with that of Taguchi et al. [24]. The compound Cu_9Zr_2 was experimentally determined in the Cu side based on analysis by EPMA, which result agrees well with previously report [17,20,21]. The Cu_5Zr_8 has not observed in the present Cu-Zr diffusion couple as previously experiments [22,24].

3.2. Growth constants of intermetallic phases

Growth constant for IMCs were determined to study the growth kinetics. For the diffusion controlled growth in a solid-to-solid diffusion couple and under the assumption of negligible nucleation time, the layer thickness of intermetallic was evaluated at each time *t* as follows,

$$k_p = \frac{Y^2}{t} \tag{1}$$

where Y is the layer thickness of intermetallic, t is annealing time, and k_p is the growth constant. The temperature dependence of the parabolic growth rate constant should follow the Arrhenius relation expressed by

$$k_p = k_o \exp(-Q/RT) \tag{2}$$

where R is the ideal gas constant, Q is the activation energy, T is the annealing temperature in Kelvin, and k_o is the pre-exponential factor, which is a constant independent of temperature. Take the logarithm on both sides of the Eq. (2), $\ln k_p$ is found to bear a linear relationship with the $\frac{1}{T}$, the slop is $-\frac{Q}{R}$ and the intercept is $\ln k_0$. Using the Eq. (1) and Eq. (2), the pre-exponential factor k_o and activation energy Q for Cu_9Zr_2 and $Cu_{51}Zr_{14}$ are presented in Table 1 with other experimental data [24]. Because of the thin thicknesses of Cu_8Zr_3, $Cu_{10}Zr_7$, CuZr and

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