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ARTICLE

Quantitative Analysis of TiB_2 Particles and Properties of Cu-TiB_2 Composite Prepared by in Situ Reaction

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Abstract: Cu-TiB_2 composites were prepared by combining in situ reaction and hot-pressing at different temperatures after ball-milling of the mixture powders of Cu, Ti and B. The reaction process of Cu-Ti-B system was discussed. By means of XRD, SEM, EDS and XPS, it is determined that TiB_2 nano-particles are generated by in situ reaction in Cu-matrix. According to the calibration curve of TiB_2 and Cu by XRD, the concrete synthesis rate of TiB_2 in Cu-matrix at different sintering temperatures by external standard method was confirmed. The results show that in certain temperature range, the higher the temperature is, the higher the synthesis rate is, and the best synthesis rate of TiB_2 is 99.27% at 1000 ℃. Cu-1.5wt%TiB_2 prepared at 1000 ℃ has the best properties, Its Vickers hardness (HV), electric conductivity (EC), flexure strength (FS), thermal expansivity (TE) and thermal conductivity (TC) at 100 ℃ are 125.68 MPa, 80.1 % IACS, 755.2 MPa, 9.3×10^{-6} K^{-1} and 260 W/(m·K), respectively.

Key words: Cu-TiB_2; in-situ; calibration curve; property

High conductivity and high strength copper alloys have many industrial applications, including welding electrodes, rail transit contact wires, IC lead frame, continuous caster material and so on[1-4]. Owing to desired combination of electrical conductivity, thermal conductivity and mechanical properties, copper matrix composites (CMCs) have attracted so much attention in recent years. Hot-pressure sintering technique has been demonstrated to be effective in preparing CMCs in the last few decades[5-8]. By mixing reinforcing phase and copper powder directly or via an interfacial design process, ex-situ reinforced CMCs were successfully prepared. Compared to the conventional ex situ reaction, in situ reaction synthesis produces superior wetting interface between particle and matrix, and the CMCs thus prepared exhibit preferable integrated performances[9]. Accordingly, the hot-press sintering has been widely applied to prepare in-situ CMCs[10-12].

Compared to the unreinforced copper, the improved properties of the CMCs mainly originate from the second-phase particles, such as borides (TiB_2, ZrB_2), carbides (TiC, SiC) and oxides (Al_2O_3)[13-15]. Among these particles, TiB_2 is deemed to be a good candidate to reinforce CMCs because of its high elastic modulus (574 GPa), high hardness value (HV:34 GPa), good thermodynamic stability and good electrical conductivity[16]. Moreover, TiB_2 particles are thermodynamically stable and can easily form through in situ reactions between titanium and boron elements in copper melt[17,18].

So far, Cu-TiB_2 composites prepared by in situ have many literatures[19,20], such as the preparation methods, microstructure and mechanical properties. The content of TiB_2 has an important effect on the properties of the composites, but there is no research related to the conversion rate of TiB_2 synthesizing in-situ reaction. In order to study the effect of second phase synthesizing in-situ reaction on the properties of composite materials, quantitative calculation for the content of the second phase is of great significance.

In this paper we decided to use elemental Ti, B and Cu as raw material to in-situ synthesize TiB_2 reinforced phase in order to get single kind of reactive products. Powders of Cu, Ti and B blended after high-energy ball-milling were

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prepared for Cu-TiB_2 composites by reactive hot-pressure sintering. The thermodynamic analysis of the Cu-Ti-B system, quantitative calculation of synthesis rate of TiB_2 particles at different sintering temperature, and the properties of the Cu-TiB_2 composites were emphatically carried out.

1 Experiment

In the present study, Cu-1.5wt%TiB_2 and Cu-15wt%TiB_2 composites were prepared in a vacuum thermocompression furnace (ZT-40-20Y) at different sintering temperatures after ball-milling the powders of Cu (purity 99.95%, 42.4 μm), Ti (purity 99.99%, 36 μm) and B (purity 99.99%, 35 μm) with a stoichiometric Ti:B of 1:2. The power mixtures were put in a stainless steel vacuum jar with alcohol. The mechanical ball-milling process was conducted for 9 h with a ball to powder ratio (BPR) of 10:1 at the milling speed of 300 r/min on planet ball grinder (QM-3SP04). Subsequently, the dried mixture powers were calcined in flowing argon atmosphere at 25 MPa in a temperature range of 600~1060 ℃ with heating rate for 10 ℃ /min and holding time for 3 h.

The sintered bulk samples were measured on X-ray diffractometer (D/MAX2500V, Japanese Neo Confucianism) with Cu Kα radiation at 40 kV and 150 mA, scan speed for 0.02 °and time per step for 0.2 s to analyze the phase of the composites and prepare for the calibration curve which can be used to calculate the synthesis rate of TiB_2 by in situ reaction. To further analyze component of the composites, XPS (ESCALAB250, U.S.A. Thermo) was conducted to identify the valence state of elements of Cu, Ti and B. The microstructure was observed and analyzed by field emission scanning electron microscopy (FE-SEM, SU8020, Hitachi) with an energy-dispersive spectroscopy (EDS) system.

All the mechanical properties were measured at room temperature. The density of solid samples was measured by Archimedes` method. The electrical resistance was measured by a double bridge method. And the Vickers hardness test was performed on the fine polished surface by HXD-1000 tester (Shanghai second optional Ltd, China) at a load of 100 g with the dwell time of 10 s. The flexural strength of bulk Cu-TiB_2 specimen with dimensions of 3 mm×8 mm×35 mm was determined by the three-point bending method at Universal testing machine. The coefficient of thermal expansion of cylinder Cu-TiB_2 specimen with diameter of 12.8 mm were measured by thermomechanical analysis unit (TMA402F3, Netzsch) and thermal conductivity of cylinder Cu-TiB_2 specimen with the diameter of 6 mm by laser flash thermal analyzer(LFA457, Netzsch). In addition, in order to analyze the strengthening mechanism of Cu-TiB_2 composite, the fracture of solid samples after three-point test was also examined by SEM.

2 Results and Discussion

2.1 Phase transformation of Cu-Ti-B system

Fig.1 shows the phase diagrams of Cu-Ti[21] and Ti-B[22], according to the mass ratio of copper to titanium and the atomic ratio of titanium to boron. the reactions will take place in the Cu-Ti-B system as follows:

$$Ti+B \rightarrow TiB \quad (1)$$

$$Ti+2B \rightarrow TiB_2 \quad (2)$$

$$Cu+4Ti \rightarrow Cu_4Ti \quad (3)$$

The relationship of reaction free enthalpy and temperature by Gibbs Helmholtz equation is as follows:

$$d\left(\frac{\Delta G_T^{\Theta}}{T}\right) = -\frac{\Delta H_T^{\Theta}}{T^2} dT \quad (4)$$

In the formula, the ΔH_T^{Θ} represents the reaction heat effect, T is for the thermodynamic temperature, ΔH_T^{Θ} can be obtained by the Kirchhoff formula:

$$d\Delta H_T^{\Theta} = \Delta C_p dT \quad (5)$$

Where ΔC_p stands for the differences of heat capacity of the reactions, C_p changes with temperature and can be approximated by the following equation:

$$C_p = a + b10^{-3}T + c10^{-5}T^{-2} + d10^{-6}T^2 + e10^8T^{-3} \quad (6)$$

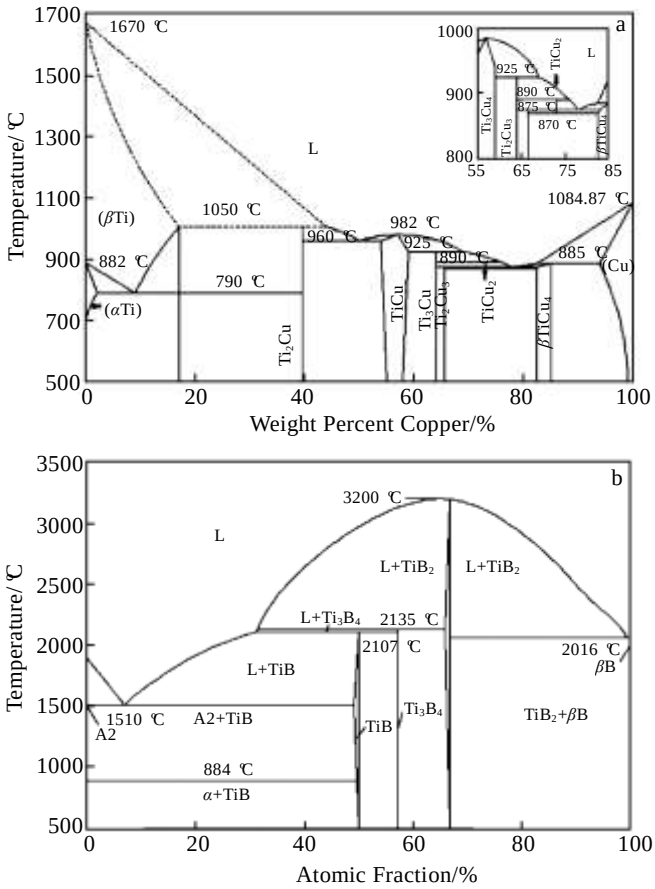

Fig.1 Phase diagrams of Cu-Ti (a) and Ti-B (b)

$$\Delta H_T^{\Theta} = \Delta aT + \frac{1}{2}\Delta b10^{-3}T^2 - \Delta c10^5 T^{-1} + \frac{1}{3}\Delta d10^{-6}T^3 - \frac{1}{2}\Delta e10^8 T^{-2} + f \quad (7)$$

$$\Delta G_T^{\Theta} = -\Delta aT\ln T - \frac{1}{2}\Delta b10^{-3}T^2 - \frac{1}{2}\Delta c10^5 T^{-1} - \frac{1}{6}\Delta d10^{-6}T^3 - \frac{1}{6}\Delta e10^8 T^{-2} + gT + f \quad (8)$$

According to consult parameters of a, b, c, d, e and the above Eqs.(4)~(8)[23], Δa, Δb, Δc, Δd, Δe, f, g can be calculated as shown in Table 1.

According to Table 1 and Eq.(8), variation of free enthalpy ΔG with temperature can be calculated in the above reaction, as shown in Fig.2.

In Fig.3, with the temperature rising, Ti and B become less and less, and TiB_2 comes into being from 850 ℃, which is in keeping with the result of above thermodynamic analysis. It also can be seen that with the sintering temperature rising, lattice constant of Cu ranges from 0.361 911 to 0.361 832, indicating that Ti and B which are original solid solution in the alpha-Cu precipitate gradually, and thus the alpha Cu basic restores to the original lattice constant (a=0.3615).

2.2 Effect of temperature on the synthesis of TiB_2

Quantitative phase analysis method which did not add standard material to the sample when tested, usually used one kind of pure phase to be tested as standard samples, needed to make a series of external standard samples and map out the working curve, is called external standard method[24].

Supposed that the mixture samples needed to be tested were composed of i phase and j phase, μ_{mi}, μ_{mj} and μ_m are integrated mass absorption coefficient of i phase, j phase and two-phase mixtures, and W_i and W_i represent the mass percentage of i phase and j phase.

$$\mu_m = W_i\mu_{mi} + W_j\mu_{mj} = W_i(\mu_{mi} - \mu_{mi}) + \mu_{mj} = W_j(\mu_{mj} - \mu_{mi}) + \mu_{mi} \quad (9)$$

The intensity of the diffraction peak of j phase in the samples to be measured is as follow:

$$I_j = C_j\frac{W_j}{\mu_m} = \frac{c_j w_j}{W_j(\mu_{mj} - \mu_{mi}) + \mu_{mi}}, C_j = CK_j/\rho_j \quad (10)$$

The intensity of the corresponding diffraction peak of j phase in the standard samples is as follows:

Table 1 Parameters of the thermodynamics computational process

T/K	Phase	Δa	Δb	Δc	Δd	Δe	f	g
298~800	Cu_4Ti	-37.45	4.73	5.56	0	0	-94296.21	-226.68
	TiB	4.11	-9.55	10.54	0	-5.21	-160443.3	27.07
	TiB_2	-21.38	17.00	46.88	-3.35	-10.42	-308329.9	-137.69
800~1155	Cu_4Ti	-37.45	4.73	5.56	0	0	-94296.21	-226.68
	TiB	10.55	-14.97	-9.52	0	0	-165922.4	73.34
	TiB_2	-8.50	6.17	6.75	-3.35	0	-319288.1	-45.15
1155~1500	TiB	12.85	-12.63	-9.52	0	0	-166712.1	89.45
	TiB_2	-6.20	8.51	6.75	-3.35	0	-320077.8	-29.04

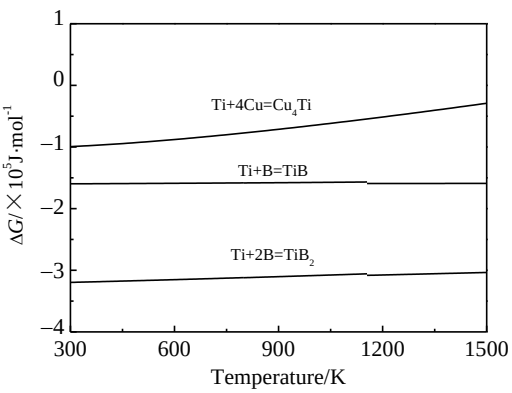

Fig.2 Relationship between ΔG and temperature

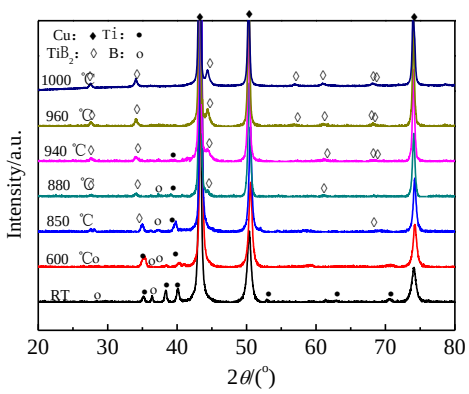

Fig.3 XRD patterns of Cu-15wt%(Ti+2B) sintered at different temperatures

$$I'_{\mathrm{j}} = C_{\mathrm{j}} \frac{1}{\mu_{\mathrm{mj}}} \tag{11}$$

It can be seen from Eq.(10) and Eq.(11)

$$\frac{I_{\mathrm{j}}}{I'_{\mathrm{j}}} = \frac{W_{\mathrm{j}}\mu_{\mathrm{mj}}}{W_{\mathrm{j}}(\mu_{\mathrm{mj}} - \mu_{\mathrm{mi}}) + \mu_{\mathrm{mi}}} \tag{12}$$

The calculation process of the mass fraction of j phase in the mixture by external standard method can be summarized as the following two steps. The first step is to make a calibration curve: prepare at least more than three different proportions of two-phase (pure j phase and i phase) mixture, test their strongest diffraction peak intensity by XRD, draw calibration curve what the abscissa is the content of j phase and longitudinal coordinate is I_j/I_i, which is equivalent to Eq.(12). The second step: in the same test conditions, measure the intensity of the corresponding diffraction peak of the mixture to be measured, find the point with the same vertical coordinates in the work curve, and the horizontal coordinate value is the mass fraction of j phase in the mixture.

So in order to calculate the synthesis rate of TiB_2, seven Cu-*x*wt%TiB_2 (*x*=2.5, 5, 10, 15, 20, 25, 30) samples with Cu powder and pure TiB_2 (99.9%, 3.8 μm) were prepared by sintering at 980 ℃. Calculate the peak intensity ratio of TiB_2 to Cu under the different mass fraction of TiB_2 by XRD as show in Table 2, then make calibration curve using the mass fraction of TiB_2(TiB_2%) as the abscissa and the peak intensity ratio of TiB_2 to Cu(I_{TiB_2}/I_{Cu}) as the ordinate as shown in Fig.4a. In addition, Cu-*y*wt%TiB_2 (*y*=2.59, 5.34, 8.23, 14.48, 21.47, 29.35, 38.29) composites were random prepared in the same conditions again, according to the above method to draw another calibration curve, as shown in Fig.4b. The two calibration curves were nearly superposition, which indicated that the curve was with high confidence level. At last, Fig.4b was chosen as the work curve to calculate the mass fraction of TiB_2 at different sintering temperatures. And the equation was gained by making calibration curve of Cu - TiB_2 as follows:

$$\frac{I_{\mathrm{a}}}{I_{\mathrm{b}}} = 0.0132 + 0.377W_{\mathrm{a}} + 0.8919W_{\mathrm{a}}^2 \tag{13}$$

where, I_a and I_b are the integrated diffraction intensity of TiB_2 (101) peak and Cu (111) peak, respectively, and W_a represents the mass percentage of TiB_2.

Fig.5 shows XRD patterns of the samples calcined in the temperature range of 940~1060 ℃ from the milled powders of Cu-15wt%(Ti+2B). Obviously, it is clear that there are Cu phase and TiB_2 second phase in the samples. By external standard method, Fig.5 was applied to calculate the mass fraction of TiB_2 in the sample. Firstly test the strongest diffraction peak intensity of TiB_2 and Cu under the above measure conditions by XRD as shown in Table 3, and then the

Table 2 Peak intensity of the mixtures of different contents of TiB_2 and Cu

TiB_2 content/ wt%	I_{TiB_2} (avg)	I_{Cu} (avg)	I_{TiB_2}/I_{Cu} (avg)
2.5	25037	1411497	0.018
5	50669	1314626	0.039
10	75367	1204749	0.063
15	100685	1124018	0.09
20	121906	1003517	0.121
25	143529	892083	0.161
30	163137	779849	0.209
2.59	29385	1363711	0.022
5.34	43501	1303119	0.033
8.23	62971	1214559	0.05
14.48	96218	1103959	0.087
21.47	127395	900106	0.14
29.35	153851	795648	0.193
38.29	184418	619615	0.298

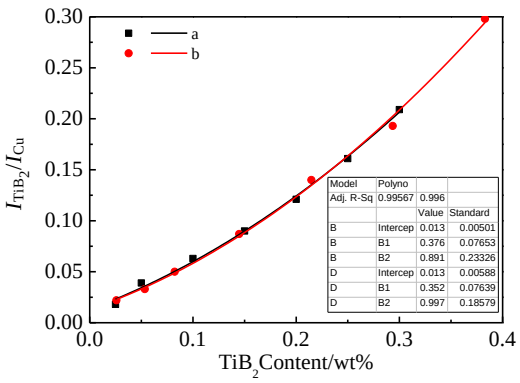

Fig.4 Work curve of TiB_2 and Cu: (a) Cu-*x*wt%TiB_2 (*x*=2.5, 5, 10, 15, 20, 25, 30); (b) Cu-*y*wt%TiB_2 (*y*=2.59, 5.34, 8.23, 14.48, 21.47, 29.35, 38, 29)

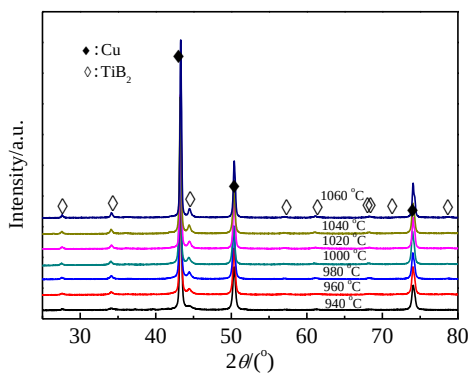

Fig.5 XRD pattern of Cu-15wt%(Ti+2B) sintered at different temperatures

Table 3 Peak intensity of Cu-TiB_2 composites

T/℃	I_{TiB_2}(avg)	I_{Cu}(avg)	I_{TiB_2}/I_{Cu}(avg)
940	54574	973437	0.05604
960	73253	1004153	0.07295
980	79066	972401	0.08131
1000	88729	995389	0.08914
1020	90343	1025925	0.08806
1040	91499	1047331	0.08736
1060	91529	1051936	0.08701

mass fraction of TiB_2 samples can be checked in Fig.5, which was the value of the horizontal coordinate corresponding to the same vertical coordinate.

From Fig.6, it can be seen that with the temperature increasing, atoms proliferated more fully and it leads to the synthesis rate of TiB_2 increasing gradually. As temperature further increases, the impact of the sintering temperature on the synthesis rate of TiB_2 changes little because of Ti and B almost completely transform into TiB_2.

2.3 Microstructure and properties of Cu-TiB_2 composites

It is observed that the TiB_2 particles synthesized by in situ in the matrix are cubic and spherical in form and the size of the TiB_2 particles ranges from 50 to 200 nm as shown in Fig.7a and Fig.7b, which is much lower than that of ex situ particles in discontinuously reinforced composites. What`s more, take measures of line-scanning on the samples, it can be seen that the peak position of Ti is the same as B elements in the black particles, which is opposite of Cu element in Fig.7c, illustrating that the TiB_2 particles are generated in the in situ reaction in keep with the result of above analysis.

Fig.8 shows XPS spectra of Cu-TiB_2 composite at 1000 ℃. The spectrum displays the spin-orbit splitting characteristic of Cu 2p, Ti 2p and B 1s levels. The intensity ratio of the Cu $2p_{3/2}$ and Cu $2p_{1/2}$ peaks is constrained to be

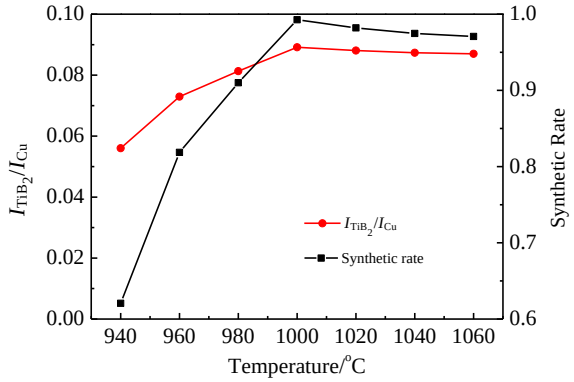

Fig.6 Synthetic rate curves of TiB_2

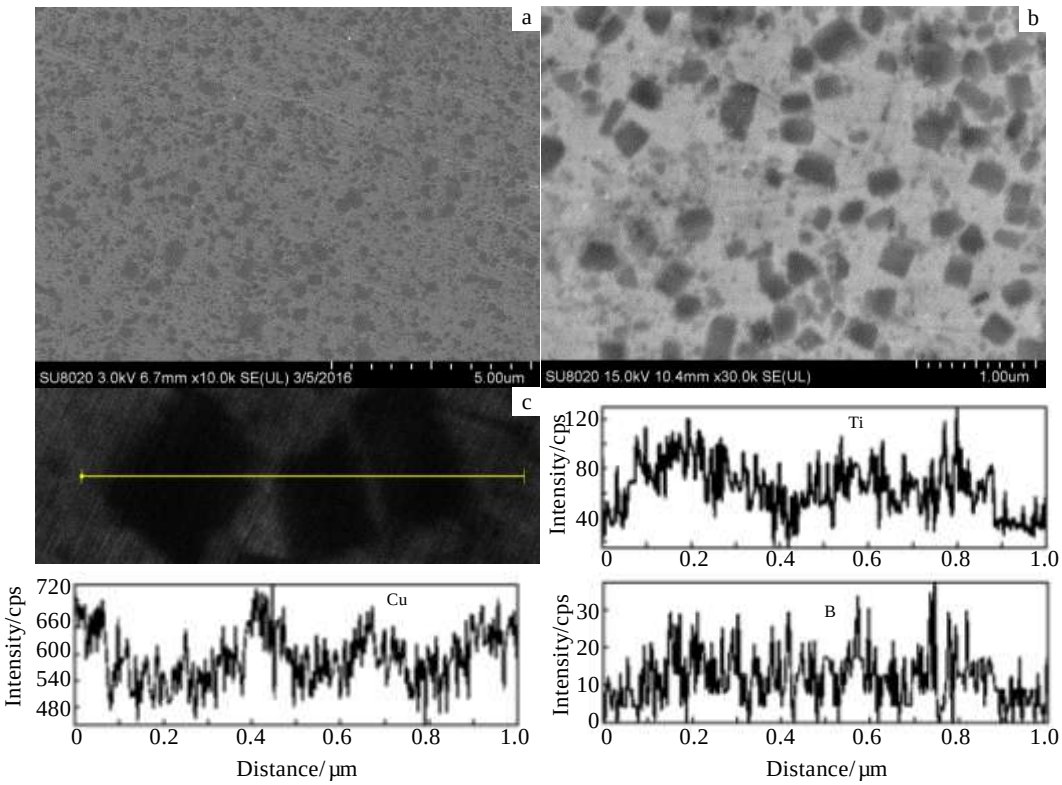

Fig.7 SEM images (a, b) and EDS element line scanning (c) of Cu-TiB_2 composite

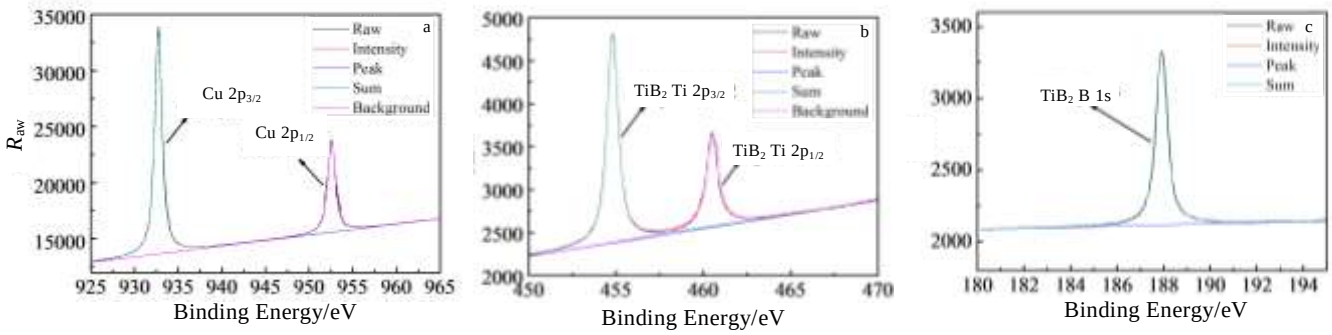

Fig.8 XPS spectra of Cu-TiB_2 composite at 1000 ℃: (a) Cu 2p, (b) Ti 2p, and (c) B ls

Table 4 Properties of Cu-TiB_2 composites prepared by different methods

Material	Methods	RD/%	Hardness, HV/MPa	EC /%IACS	TS /MPa	FS /MPa	TE /$\times10^{-6}$ K^{-1}	TC /W (m·K)$^{-1}$
Cu	-	99.8	60	99.9	206	-	17.5	397
Cu-1.5%TiB_2	Solid-solid(HP)	98.28	125.68	80.1	560	755.2	9.3	260
Cu-2%TiB_2[11]	Solid-solid(MA)	-	-	78.4	545	-	-	-
Cu-0.5%TiB_2[29]	Solid-liquid(RSD)	-	-	63.5	410	-	-	-
Cu-2.6%TiB_2[30]	Liquid-liquid(DBM)	-	-	76	675	-	-	-

2:1 during the fitting procedure and so does Ti. Linear background is used during the fitting procedure. The peak of Ti 2p at about 454.7 and 460.4 eV as reported for TiB_2[25,26]. And only one peak of B can be fitted at around 187.9 eV as reported for TiB_2[27,28]. It can be seen that Cu element without forming the other compound, Ti element and B element almost exist in the form of TiB_2. The result is the same as XRD analysis, which further proved that the Ti powder and B powder synthesized TiB_2 in situ.

From Table 4, it can been seen that Cu-TiB_2 composite prepared by hot-pressing(HP) at 1000 ℃ contrast with pure copper significantly improves the stability and increases hardness, while the electrical conductivity decreases slightly. And its electric conductivity and tensile strengthen are higher than that of Cu-TiB_2 composites manufactured by mechanical alloying (MA) and reaction spray deposition forming process (RSD). Cu-2wt%TiB_2 prepared by MA requires long time for 25 h though with low temperature of 890 ℃ [11]. Cu-0.5wt%TiB_2 prepared by RSD demanded high temperature of 1400~1500 ℃; furthermore, it had segregation and incompleted reaction[29]. Although Cu-5vol%TiB_2 (equal to Cu-2.6wt%TiB_2) prepared by double beam melts(DBM) had relatively good properties, it not only needed to react at high temperature, but also the size and distribution of TiB_2 were not easy to control[30].

3 Conclusions

1) By the external standard method, TiB_2 nano-particles generated by in situ reaction in Cu-matrix are determined. The amount of TiB_2 in the samples is calculated by the ratios of the peak intensities of TiB_2 and Cu obtained from XRD studies. As sintering temperature increases, the synthesis rate of TiB_2 becomes higher, and the best synthesis rate is up 99.27% at 1000 ℃.

2) By composite technology of in situ reaction and hot-pressure sintering, high-performance composites Cu-TiB_2 is produced, and the size of the enhancement TiB_2 particle is about 50~200 nm, which is of uniform distribution without segregation.

3) The best comprehensive performance of Cu-1.5wt% TiB_2 composites is obtained by hot-pressing sintering at 1000 ℃, including the density 98.28%, conductivity 80.1%IACS, micro Vickers hardness (HV) 125.68 MPa, and bending strength 755.2 MPa, thermal expansion coefficient 9.3×10^{-6} K^{-1} at 100 ℃ and thermal conductivity 260 W (m·K)$^{-1}$ at 100 ℃.

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原位合成 TiB_2 的定量计算及 Cu-TiB_2 复合材料性能的研究

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摘　要：以 Ti 粉、B 粉和 Cu 粉为原材料，球磨后，采用热压法原位合成 Cu-15%TiB_2 (质量分数) 复合材料。并详细讨论了 Cu-Ti-B 体系的反应过程。通过 XRD、SEM、EDS、XPS 等手段，确定了 Ti 和 B 在 Cu 基体中原位合成了 TiB_2，并利用 XRD 制作 TiB_2 和 Cu 的定标曲线，采用外标法计算出不同烧结温度下 TiB_2 的合成率。结果表明，在一定的温度范围内，温度越高，合成率越高，在 1000 ℃时 TiB_2 的合成率可达 99.27%。并测试 Cu-1.5%TiB_2 块状试样的维氏硬度，电导率和三点弯曲强度，分别为 125.68 MPa、80.1% IACS 和 755.2 MPa，在 100 ℃时的热膨胀系数和导热系数分别为 9.3×10^{-6} K^{-1} 和 260 W/(m·K)。

关键词：Cu-TiB_2；原位；定标曲线；性能

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