Original Research

Joining of Si₃N₄ ceramic using PdCo(NiSiB)–V system brazing filler alloy and interfacial reactions

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Abstract

The wettability of V-active PdCo-based alloys on Si_3N_4 ceramic was studied with the sessile drop method. And the alloy of Pd50.0–Co33.7–Ni4.0–Si2.0–B0.7–V9.6 (wt%), was developed for Si_3N_4 ceramic joining in the present investigation. The rapidly-solidified brazing foils were fabricated by the alloy Pd50.0–Co33.7–Ni4.0–Si2.0–B0.7–V9.6. The average room-temperature three-point bend strength of the Si_3N_4/Si_3N_4 joints brazed at 1453 K for 10 min was 205.6 MPa, and the newly developed braze gives joint strengths of 210.9 MPa, 206.6 MPa and 80.2 MPa at high temperatures of 973 K, 1073 K and 1173 K respectively. The interfacial reaction products in the Si_3N_4/Si_3N_4 joint brazed at 1453 K for 10 min were identified to be VN and Pd_2Si by XRD analysis. Based on the XEDS analysis result, the residual brazing alloy existing at the central part of the joint was verified as Co-rich phases, in which the concentration of element Pd was high up to 18.0–19.1 at%. The mechanism of the interfacial reactions was discussed. Pd should be a good choice as useful alloying element in newer high-temperature braze candidates for the joining of Si-based ceramics.
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Keywords: Si_3N_4; Wettability; Joining; Interfaces; Reaction

1. Introduction

It is well known that advanced ceramic materials such as Si_3N_4 and SiC have a high potential for high-temperature application. However, the usefulness of most engineering ceramics is often limited by ceramic joining techniques. Requirements for these ceramic joints include sufficient mechanical strength and the possibility to withstand high temperatures. It has been recognized that brazing technique is one of reliable techniques for ceramic joining, and since 1990s the development of new high-temperature brazes for the joining of ceramic to ceramic and ceramic to metal had become a research focus.

The wettability of traditional brazes on Si_3N_4 is not so good due to the fact that there exists a great difference in physical and chemical properties between ceramic and metal. Therefore, some active elements such as Ti, Zr, Hf, Cr and V are often added into brazing alloys to induce chemical reactions at the interface between the ceramic and the metal, and the resulted interfacial reaction layer is necessary for the joining of ceramic to metal.

Hadian et al. used Ni–Cr–Si system alloy as brazing filler for Si_3N_4/Si_3N_4 joining [1], in which Cr was used as the active element, but four-point bend strength of the corresponding joints at room temperature was low, only 118 MPa. Reichel et al. made an attempt to join Si_3N_4 ceramic by using Ti-active Co-based brazing alloy [2], but the result was not satisfactory.

Some other researchers designed V-active brazing alloys containing noble metals such as Au, aiming at that the brazed ceramic joint had a high service temperature of 923 K, and some good results were indeed achieved for Si_3N_4 joining, for example, Paulasto et al. used Au–36.6Ni–4.7V–1Mo (at%) alloy as brazing filler, and four-point bend strengths of the Si_3N_4/Si_3N_4 joints are

high up to 345–393 MPa at room temperature [3]. However, the corresponding high-temperature joint strength is not so high, for example, at test temperatures of 973 K and 1073 K, the joint strength shows the values of 150 MPa and 80–100 MPa respectively. Furthermore, it is well known that Au is an expensive metal. Sun et al reported the joining of Si_3N_4 ceramics using a Au–Ni–Pd–V filler alloy at 1423 K for 60 min, and the bend strengths of the achieved joints were 264.4 MPa at room temperature and 214.2 MPa at 1073 K respectively [4], but the Au content in this filler alloy was still high up to 54.2 at% (or 78.7 wt%), and the joint strength at 1173 K decreased sharply to only 13 MPa.

Therefore, searching for cheaper brazes with an improved high-temperature joint strength for Si_3N_4 joining, should be of great significance.

In general so-called active braze for ceramic joining should be composed of a base brazing metal (or alloy) and an active element. The difficulty in developing new high temperature active brazes for Si_3N_4 joining lies in finding a suitable base brazing alloy, as this base brazing alloy has to meet the following three requirements: firstly, the base brazing alloy itself should exhibit an excellent high-temperature properties, secondly, its liquidus temperature should not be too high, and the third, most important, the chosen active element must exhibit its active effect very well in this base brazing alloy during the brazing process. Here, exhibiting active effect very well means inducing suitable reactions between the active braze and the joined ceramic. Undue reactions between them will conversely be harmful to the ceramic joining. In the previous research work the authors studied the wettability of V-active Ni(Co)-based alloys on Si_3N_4, and the reactions between the V-active alloys and the Si_3N_4 at 1553 K were intensive, that is, the thickness of the reaction layer was high up to hundreds of micrometers [5]. Such undue reactions induced a monotonously fracture at the interface between the Si_3N_4 and the interfacial reaction layer for all of the specimens. This also signifies that V-active Ni(Co)-based alloys have no high potential as brazing filler candidates for Si_3N_4 joining.

On the other hand, Sera et al studied the Si_3N_4 joining by using V-active Cu-based alloy brazes and the maximum shear strength of the corresponding joints at room temperature is 280 MPa [6]. Ito et al also reported that the joining of Si_3N_4 ceramic to metal Mo by using vanadium foil, and sound joint was achieved [7]. However, so far, the development of new high-temperature V-active brazes for Si_3N_4 joining and the understanding of the interfacial reactions are still far insufficient.

Presently the price of metal Pd is much lower than that of Au. In addition, Pd possesses good thermal and oxidation resistance, and Pd-based filler metals have been primarily investigated, including Pd58.2–Ni38.3–Ti3.0 (wt%) filler metal and Pd/CuTi multilayer interlayers [8,9]. Therefore it is believed that PdCo-based alloys should have good high-temperature properties. Taking the above two factors into account, in the present paper the authors proposed V-active PdCo-based brazing filler alloys for Si_3N_4 joining, and we put emphasis on the high temperature strengths of the brazed Si_3N_4 joints and the involved reaction mechanism.

2. Experimental

Firstly we designed four composition Pd–Co–V alloys, in which the weight ratio of Pd to Co is fixed at 3:2. Their compositions are Pd60–Co40, Pd57–Co38–V5, Pd54–Co36–V10, and Pd48–Co32–V20. The contact angles of these Pd–Co–V alloys on Si_3N_4 during heating in a vacuum condition were measured with the sessile drop method. The Pd–Co–V alloys were prepared monotonously by the following process: high-purity (> 99.5%) powders (− 200 mesh) of Pd, Co and V were weighed in the designed ratio and blended mechanically, and then the mixed powder was pressed by a fine mold into a cylindrical pellet with a diameter of 4 mm. The mass of the pellet was in the range of 80–100 mg.

The wetting experiment was conducted in a vacuum brazing furnace. Hot-press sintered Si_3N_4 ceramic with about 12 wt% Al_2O_3 and Y_2O_3 as sintering activator was used as material substrate in the wetting experiment, with a size of $11 \times 11 \times 3$ mm^3. The ceramic surface was polished by 1000# SiC sand paper and subsequent diamond paste with a size of 1.0 μm so that it showed a mirror-bright surface. The Pd–Co–V powder pellets were put on the Si_3N_4 plates separately, and then they were put into the vacuum furnace. Subsequently the furnace was evacuated to 2.0×10^{-3} Pa by a mechanical rotary pump and an oil diffusion pump, and the samples were heated to 1523 K at a heating rate of 8–10 K/min. During the high-temperature period in the wetting experiment the vacuum of the furnace was kept at $3.0–5.0 \times 10^{-3}$ Pa. After being held at the high temperature for 30 min the samples were cooled to room temperature at a cooling rate of about 10 K/min. Finally the samples were taken out of the furnace and their contact angles were measured with an optical microscope at room temperature.

Then, a novel composition of Pd–Co–V alloy, Pd50.0–Co33.7–Ni4.0–Si2.0–B0.7–V9.6 (wt%) was designed, in which elements Si and B were used as temperature depressant. The PdCo(NiSiB)–V alloy was fabricated into brazing foils by a rapidly-solidifying technique, and the acquired brazing foils were about 45 μm thick and 12 mm wide (shown in Fig. 1). The wetting experiment under the previously-described condition was also performed for this alloy in the form of brazing foils. The liquidus temperature of the PdCo(NiSiB)–V filler
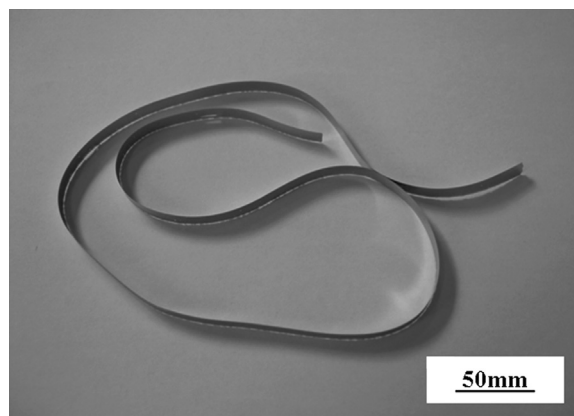

Fig. 1. Morphology of the rapidly-solidified PdCo(NiSiB)–V brazing foils.

alloy was measured by a differential thermal analysis (DTA) method.

The PdCo(NiSiB)–V filler foils were used in the subsequent joining experiment. The samples to be brazed were fixed in a specially designed graphite jig to form a butt joint. The size of the Si_3N_4 bars to be joined was $3 \times 4 \times 20$ mm^3.

Before joining, the filler foils and the Si_3N_4 samples to be joined were ultrasonically cleaned in acetone and dried by air blowing. Four brazing temperatures were chosen, and brazing time was fixed at 10 min. During the brazing experiment the vacuum was kept between 3.0×10^{-3} Pa and 7.0×10^{-3} Pa. After brazing the cooling rate was 15 K/min down to 673 K, then about 3 K/min to room temperature. The strength of the butt joints was determined at room temperature and at high temperatures by a three-point bend test. Five specimens were tested for each experimental condition.

A JEOL-5600LV (Akishima city, Japan) type scanning electron microscope (SEM) was used for microstructure observation of the brazed joint. Furthermore, an Oxford Inca type X-ray energy-dispersive spectrometer (XEDS) was used for semi-quantitative composition analysis of some typical microzones at the interface. The samples used in this analysis were coated with a thin layer of gold and thus element Au with several atomic percentages was detectable at the interface.

For better phase identification of the Si_3N_4/Si_3N_4 joint, X-ray diffraction (XRD) analysis was performed on a simulated specimen of the Si_3N_4/Si_3N_4 joint brazed at 1453 K/10 min. The simulated specimen was prepared by the following process: 0.15-mm-thickness alloy plate was prepared by spark cutting and surface polishing from a button ingot of the PdCo(NiSiB)–V brazing alloy, and it was placed on a Si_3N_4 sample with a size of $8 \times 8 \times 2$ mm^3; then, they were put in a vacuum furnace, heated to 1453 K and held there for 10 min, followed by a slow cooling. Subsequently, gradual polishing was conducted through the reaction zone on the sample surface, and XRD analysis was carried out successively on the surface step by step.

3. Results and discussion

3.1. Wettability of Pd–Co–V system alloys on Si_3N_4 ceramic

The results show that, after being held at 1523 K for 30 min, Pd60–Co40, Pd57–Co38–V5, Pd54–Co36–V10 and Pd48–Co32–V20 alloys melted completely but they monotonously exhibited non-wetting and no adhesion with the Si_3N_4 (Fig. 2 (a)–(d)), e.g., Pd60–Co40 alloy gave a high contact angle of 134°. For Pd48–Co32–V20 alloy, its surface even showed yellowish-brown color. This phenomenon is believed to be induced by slight oxidation during the heating process as the concentration of active element V in the alloy is high up to 20 wt% and V is used in the form of fine powders.

The newly designed Pd50.0–Co33.7–Ni4.0–Si2.0–B0.7–V9.6 (wt%) alloy exhibited a low contact angle of 31° on the Si_3N_4 ceramic (shown in Fig. 2(e)) and sound bonding was achieved at the interface between the alloy sample and the ceramic.

Generally, there is a close relationship between wettability and surface tension of liquids. So far, some data for the surface tension of liquid metals and binary alloys such as Ni–Si alloy [10] and Co-based alloy [11] have been reported. However, the surface tension of such a complex alloy of Pd50.0–Co33.7–Ni4.0–Si2.0–B0.7–V9.6 (wt%) is not found from the current publications. A thorough calculation of its surface tension is necessary to clarify the reason why those Pd–Co–V system alloys do not wet with Si_3N_4 ceramic whereas the wettability of Pd50.0–Co33.7–Ni4.0–Si2.0–B0.7–V9.6 (wt%) alloy is remarkably improved.

3.2. Joint strength at room temperature and joint microstructure

The DTA analysis result shows that the solidus and liquidus temperatures of the PdCo(NiSiB)–V brazing alloy are 1400.9 K and 1408.7 K respectively. Then four brazing temperatures of 1433 K, 1453 K, 1473 K and 1493 K were chosen. As shown in Fig. 3, the Si_3N_4/Si_3N_4 joints brazed at 1433 K for 10 min
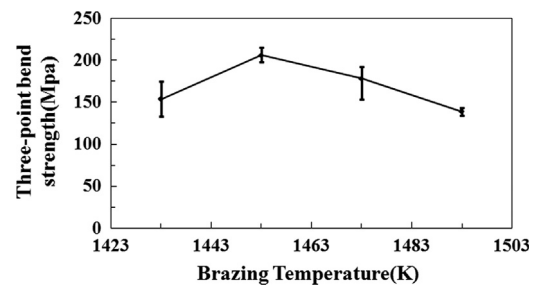
Fig. 3. Effect of brazing temperature on joint strength (Brazing filler: PdCo (NiSiB)–V foil; brazing time: 10 min).
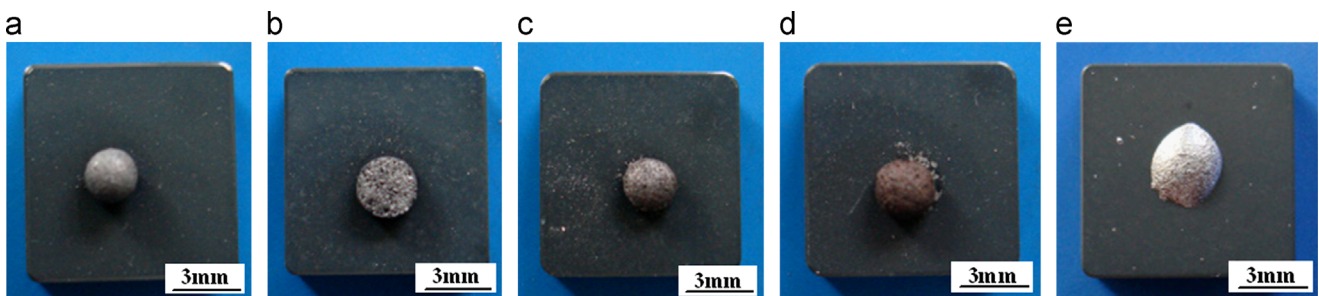
Fig. 2. Morphologies of sessile drop for different alloys on Si_3N_4 after being held at 1523 K for 30 min. (a) Pd60–Co40; (b) Pd57–Co38–V5; (c) Pd54–Co36–V10; (d) Pd48–Co32–V20; and (e) Pd50.0–Co33.7–Ni4.0–Si2.0–B0.7–V9.6.
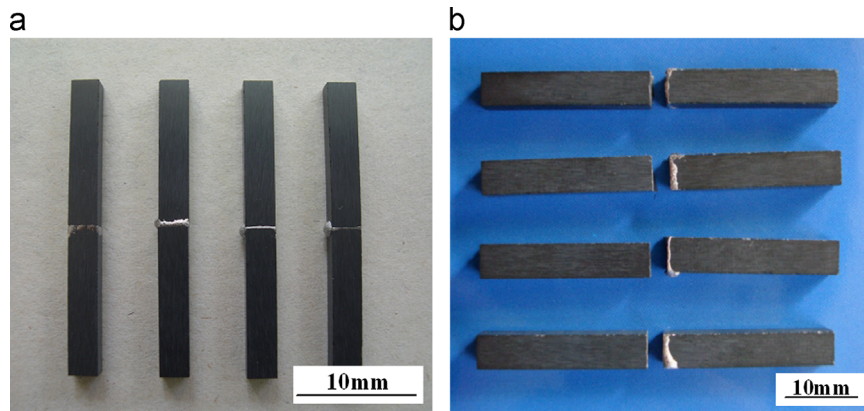
Fig. 4. Brazed Si_3N_4/Si_3N_4 joints (brazing temperature: 1453 K; brazing time: 10 min). (a) as-brazed and (b) after three-point bend test.
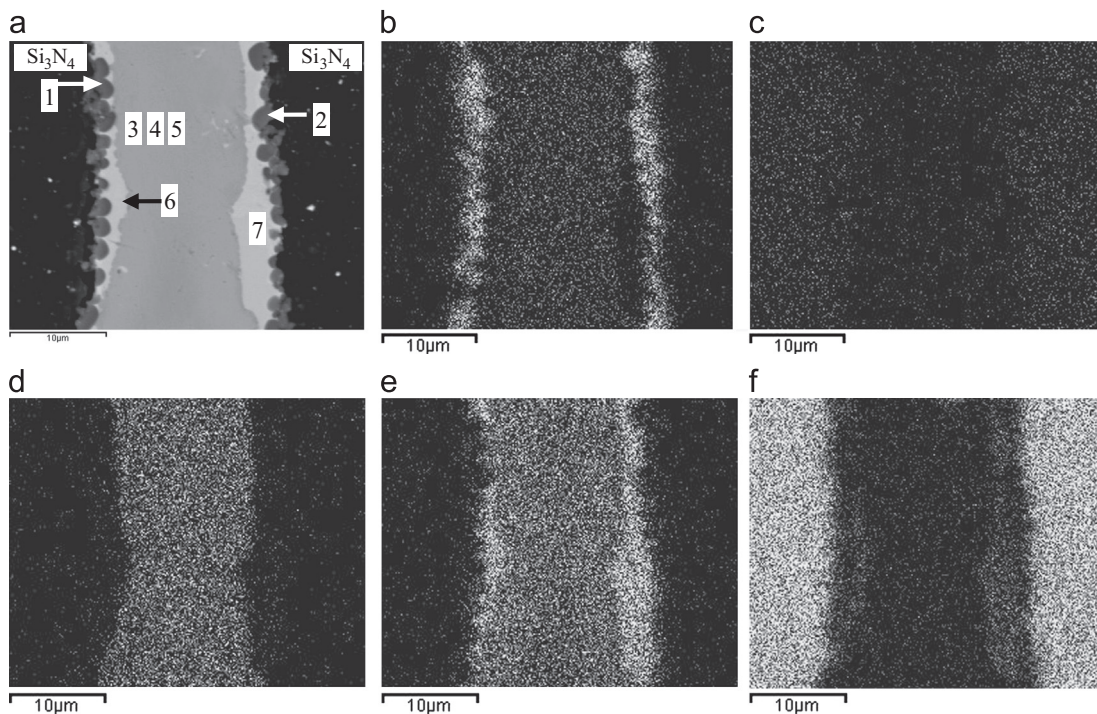

Fig. 5. Back scattered electron image (a) of Si_3N_4/Si_3N_4 joint (1453 K/10 min) and element distribution maps of V(b), N(c),Co(d), Pd (e), and Si(f).

exhibited an average three-point bend strength of 153.5 MPa at room temperature, and the average joint strength was increased to 205.6 MPa when the brazing temperature was increased to 1453 K. Sound joints brazed under the condition of 1453 K/ 10 min are shown in Fig. 4(a). The results of the joint bend test indicate that the cracks initiated at the brazed interface and propagated into the adjacent ceramic matrix (shown in Fig. 4(b)). When the brazing temperature was further increased to 1473 K and further to 1493 K, the joint strength was continuously decreased.

Fig. 5(a) presents back scattered electron image of the Si_3N_4/Si_3N_4 joint brazed with the PdCo(NiSiB)–V filler foils at 1453 K for 10 min. Two greyish quasi-continuous reaction bands (marked microzones "1" and "2" in Fig. 5(a)), with a thickness of 2–3 μm, are visible at the two interfaces close to Si_3N_4. Undoutedly, the active element V segregated strongly to

the surface of the Si_3N_4 ceramic, and according to the XEDS analysis results shown in Table 1, the corresponding reaction product should be V–N compound (Fig. 5(b) and (c)). Based on the XEDS analysis result, the residual brazing alloy existing at the central part of the joint was verified as Co-rich phases (shown in microzones "3", "4" and "5" in Fig. 5(a) and (d)). And then, two continuous white reaction layers (microzones "6" and "7" in Fig. 5(a)) formed at the interfaces between the V–N layer and the residual brazing alloy. The XEDS analysis results (Table 1) together with the element distribution maps of Pd and Si (Fig. 5(e) and (f)) indicate the presence of Pd_2Si compound there. It is clear that the concentrations of elements Pd and V in the residual brazing alloy are evidently lower than those in the original brazing alloy, resulting from the fact that the formation of previously-described V–N and Pd_2Si reaction layers would have consumed some Pd and V atoms.

Table 1
XEDS analysis results of microzones marked in Fig. 5(a).

Microzone	Concentration (at%)							Main Phase
	N	Si	V	Co	Ni	Pd	Au	
1	44.28	3.42	44.79	0.76	–	5.26	1.49	V–N
2	46.79	2.37	44.54	0.98	–	3.79	1.53	V–N
3	–	4.10	9.38	62.89	1.62	18.83	3.18	Co-rich phase
4	–	2.84	7.81	64.16	2.33	19.10	3.76	Co-rich phase
5	–	2.92	6.77	66.09	2.90	18.02	3.30	Co-rich phase
6	–	30.02	–	2.46	0.67	64.49	2.36	Pd_2Si
7	–	29.95	–	1.78	–	64.97	3.30	Pd_2Si

3.3. Interfacial reactions

In fact, the chemical reactions at high temperatures between Si_3N_4 and element V are complex, so that different reaction products have been reported in some references under different experimental conditions. For example, Paulasto et al. reported the formation of VN_x under the condition of 1273 K/30 min in Ar at Si_3N_4/Au–Ni–V–Mo interface, and that no V–silicides were found at the interface [3]. However, conversely, in the system of Si_3N_4/V/Mo under a bonding condition of 1328 K/5.4 ks/20 MPa, only V_3Si formed at the interface, and no vanadium nitride was found [7]. Moreover, in this study, when the bonding temperature increased to 1523 K, in addition to V_3Si, another silicide, V_5Si_3 formed at the interface.

But, Maeda et al. reported that in the system of Si_3N_4/V/Si_3N_4 under a bonding condition of 1473 K/3.6 ks/140 MPa, the compounds of V_3Si and V_2N were concurrently formed at the interface, and with the increase of bonding temperature and the prolonging of bonding time, besides V_3Si and V_2N, new reaction products of $V_5Si_3N_{1-x}$ and VN appeared in the joint [12]. In fact, in an earlier study Schuster et al. [13] pointed out that at 1273 K a ternary phase $V_5Si_3N_{1-x}$ was in equilibrium with Si_3N_4, V_9N_4, V_3Si, V_5Si_3, V_6Si_5 and VSi_2.

Additionally, according to the estimation of Sera et al. [6], after brazing at 1423 K for 0.3 ks–0.9 ks using Al–V–Ni–Cu filler metal, reaction products of V–Ni–Si, V_2N and VN should be formed at the interface close to Si_3N_4. In the authors′ previous study [5], intensive interfacial reactions were demonstrated under the heating condition of 1553 K/120 min for the reaction system of Si_3N_4/Ni–V alloy, and the interfacial reaction products were composed of V_3Ni_2Si, V_6Si_5 and VN. In the above-two-mentioned cases, the element Ni also participated in the interfacial reactions.

However, in the present study, it appeared that at the brazing temperature of 1453 K for 10 min the elements Co and Ni in the brazing alloy did not participate in the interfacial reactions at all. Furthermore, V–Si compounds were not detectable at the interface. The XRD results shown in Fig. 6 indicate that the interfacial reaction products are composed of VN and Pd_2Si.

According to the thermodynamic data [14], the standard Gibbs energy of formation of VN at 1500 K is −91 kJ/mol. While, the free energy of formation of Si_3N_4 can be represented by the following expression [15]:

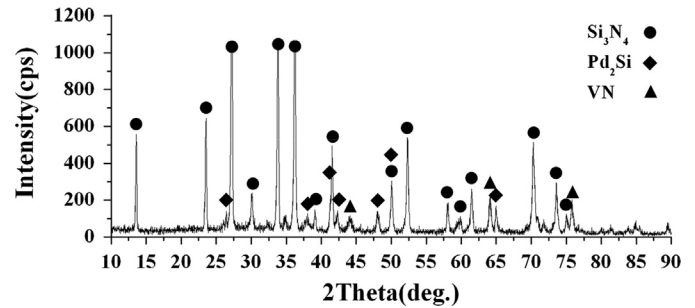$$\Delta G^o_{F(Si_3N_4)} = -396480 + 206.64T \tag{1}$$

Fig. 6. X-ray diffraction pattern for the Si_3N_4/PdCo(NiSiB)–V braze interface.

Table 2
The dissolution enthalpies of Pd, Co, Ni and V in Si melt [16].

Pd (kJ/mol)	Co (kJ/mol)	Ni (kJ/mol)	V (kJ/mol)
−158	−78	−85	−121

Then, from the above equation, $\Delta G^o_{F(Si_3N_4)}$ at 1500 K should be −86.52 kJ/mol. This means that, at 1500 K Si_3N_4 is not so thermodynamically stable as VN. Therefore, at high temperatures active element V would diffuse from the brazing alloy to the surface of Si_3N_4 and react with Si_3N_4 through the following formula:

$$Si_3N_4 + 4V = 3Si + 4VN \tag{2}$$

Then, usually, at high temperatures the Si atoms released from Si_3N_4 would diffuse into the brazing alloy where they would further react with the elements Pd, Co, Ni and V in the alloy, to precipitate Pd–Si, Co–Si, Ni–Si and V–Si compounds. However, in our experiment, among the previous four kinds of silicides, only Pd–silicide was detectable at the interface (microzones "6" and "7" in Fig. 5(a)). The thermodynamic data [16] (Table 2) signify that the combining capacity of Pd with Si should be the strongest among the four elements. On the other hand, Pd is the main element in the PdCo(NiSiB)–V brazing alloy. And, in particular, Pd_2Si is the most refractory compound in the Pd–Si system, with a melting point of 1658 K [17]. Therefore it is not strange that, for the short reaction time of 10 min, only Pd_2Si compound formed at the interface.

Then, the reaction for the formation of silicide at the interface can be expressed by the following equation:

$$2Si + Pd = Pd_2Si \tag{3}$$

Therefore, the overall reactions occurred at the interfaces between the PdCo(NiSiB)–V filler alloy and the Si_3N_4 during the brazing process can be simply expressed by the following equation:

$$Si_3N_4 + 4V + 6Pd = 4VN + 3Pd_2Si \tag{4}$$
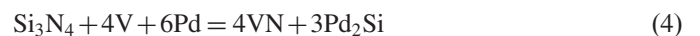
It should be noted that, the concentration of element Si in the residual brazing alloy is a little lower than the original brazing alloy (Table 1), indicating that the needed Si atoms for the formation of Pd_2Si reaction layers should actually come not only from the Si_3N_4 but also from the brazing alloy.

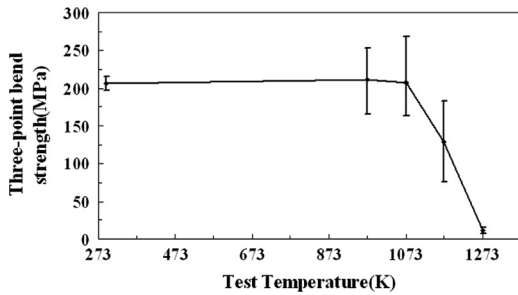As the residual brazing alloy is concerned, it is approximately composed of 62.9–66.1 at% Co, 18.0–19.1 at% Pd and

Fig. 7. Effect of test temperature on three-point bend strength of the Si₃N₄/Si₃N₄ joints brazed with PdCo(NiSiB)–V filler metal.

6.8–9.4 at% V (Table 1). From the phase diagram of Co–Pd alloy, the two elements can mix each other from 0 to 100%, and furthermore, element V with a concentration of greatly higher than 10 at% is dissoluble in Co–V or Pd–V binary alloy [18]. Once again, the formation of VN and Pd_2Si reaction layers during the brazing process have consumed some Pd and V atoms, and so it is reasonable to form these Co-rich phases at the central part of the joint.

3.4. Joint strength at high temperatures

Fig. 7 presents the strength change of the Si_3N_4/Si_3N_4 joints brazed under the optimum brazing condition with test temperature. The joints exhibit average three-point bend strength of 205.6 MPa at room temperature, and the strengths at 973 K, 1073 K, 1173 K and 1273 K are 210.9 MPa, 206.6 MPa, 80.2 MPa and 11.9 MPa respectively. This means that, the room-temperature joint strength can be retained up to 973–1073 K, and the joint strength at 1173 K, 80.2 MPa, is about 40% of that at room temperature. Evidently, from this point of view, the newly-developed PdCo(Ni, Si, B)–V brazing alloy, at least, compared with the previously reported Au–36.6Ni–4.7V–1Mo filler, indeed gives an improved high-temperature strength for Si_3N_4/Si_3N_4 joint. It is well known that Pd possesses good thermal and oxidation resistance. In the present study, on one hand, the concentration of element Pd in the residual brazing alloy at the central part of the joint brazed with the PdCo(Ni, Si, B)–V filler metal is high up to 18.0–19.1 at% (Fig. 5 and Table 1). On the other hand, two continuous Pd_2Si reaction layers have been formed at the interfaces between the VN reaction layer and the residual brazing alloy. It is reasonable to deduce that the high concentration of element Pd in the residual brazing alloy and the refractory Pd_2Si compounds at the interfaces should have contributed to the improved high-temperature joint strength.

In future, when we make further attempts to search for newer high temperature braze candidates for the joining of Si-based ceramics, Pd should be a good choice as useful alloying element [19].

4. Conclusions

(1) After being held at 1523 K for 30 min, the four composition alloys of Pd60–Co40, Pd57–Co38–V5, Pd54–Co36–V10 and Pd48–Co32–V20 (in wt%), prepared by pressing mixed high-purity powders into a cylindrical pellet, monotonously exhibited non-wetting and no adhesion with the Si_3N_4. Then, we designed a novel composition of Pd–Co–V system alloy, Pd50.0–Co33.7–Ni4.0–Si2.0–B0.7–V9.6 (wt %), and this alloy was fabricated into rapidly-solidified filler foils. After being held at 1523 K for 30 min, this filler alloy exhibited a low contact angle of 31° on the Si_3N_4 ceramic and a sound bonding was achieved at the interface.

(2) The average room-temperature three-point bend strength of the Si_3N_4/Si_3N_4 joints brazed with the newly developed brazing filler metal at 1453 K for 10 min is 205.6 MPa, and the newly developed braze gives joint strengths of 210.9 MPa, 206.6 MPa and 80.2 MPa at high temperatures of 973 K, 1073 K and 1173 K respectively.

(3) The interfacial reaction products in the Si_3N_4/Si_3N_4 joint brazed at 1453 K for 10 min were identified to be VN and Pd_2Si, and the residual brazing alloy existing at the central part of the joint is verified as Co-rich phases, in which the concentration of element Pd is high up to 18.0–19.1 at%. The Co–Pd-rich phases in the residual brazing alloy together with the refractory Pd_2Si compounds formed at the interfaces should have contributed to the improved high-temperature strengths of the Si_3N_4/Si_3N_4 joints.

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