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ARTICLE

Effects of Ti Activity on Mechanical Properties and Microstructures of Al₂O₃/Ag-Cu-Ti/Fe-Ni-Co Brazed Joints

Xin Chenglai, Li Ning, Yan Jiazhen, Cao Yongtong

Sichuan University, Chengdu 610065, China

Abstract: Al₂O₃ ceramic/Fe-Ni-Co joints were achieved using Ag-Cu-Ti filler alloy to investigate the dependence of the joint microstructure and mechanical properties on the Ti content. Scanning electron microscope (SEM), energy dispersive X-ray spectrometer (EDS), X-ray diffractometer (XRD) and electronic universal testing machine were used to characterize the microstructure and mechanical properties of the joints. The results show that the increase of Ti content can obviously intensify the interaction between Al₂O₃ and Ag-Cu-Ti. A layer of Ti-Al and Ti-O products is observed at the interface of Al₂O₃/Ag-Cu-Ti. The tensile strength of Al₂O₃/Ag-Cu-Ti/Fe-Ni-Co joint increases as the Ti content increases from 2 wt% to 8 wt%, and the tensile strength of the brazing joint reaches the maximum value of 78 MPa. Good reaction layer is formed by metallurgical reaction at the interface of Al₂O₃/Ag-Cu-4Ti, and the typical microstructure is Al₂O₃/TiAl+Ti₃O₅/NiTi+Cu₃Ti+Ag(s,s)/Ag(s,s)+Cu(s,s)+(Cu,Ni)/ Fe-Ni-Co when brazed at 890 °C for 5 min. Compared with AgCu4Ti filler, the microstructure of the joints changes slightly when addition of the active element Ti to 8 wt% except for the increase of the thickness of reaction layer adjacent to Al₂O₃ ceramic, which is accompanied by the formation of TiO and Ti₃Al in the Al₂O₃/Ag-Cu-Ti interface.

Key words: Al₂O₃ ceramic; Fe-Ni-Co; Ti activity; mechanical property; microstructure

Alumina (Al₂O₃) ceramics are widely used in electronic, aerospace, nuclear and automotive industries due to a combination of the excellent elevated temperature strength and resistance to corrosion, abrasion^[1,2]. However, the inherent brittleness and hardness of Al₂O₃ ceramics restrict their application in structural applications. Therefore in order to achieve their excellent properties in practical applications, Al₂O₃ ceramics are usually brazed to metals^[1-3], in which Fe-Ni-Co metal alloy is just one of candidates with close CTE to Al₂O₃. The most frequently used methods for joining ceramics to metal is brazing. Currently, there are two commonly used brazing techniques in industry to join Al₂O₃ ceramics to metal: one is the method of surface metallization with subsequent Ni plating of ceramic surface before brazing, and the other is active brazing by active filler alloy containing a small amount of Ti, Zr or Hf elements. The method of surface metallization of ceramic is a multi-step process, which is achieved by modifying the bonding surfaces of the ceramics

to be wettable for the conventional alloys. Nevertheless, the inadequacies of complex process and time-consuming limit its far-ranging applications in engineering. To simplify operation process and shorten the production cycle, an active brazing method with an active filler alloy to wet the surface of ceramics has been appreciated, which is achieved by the formation of an intermediate reaction layer.

A series of Titanium containing active brazing alloys are used to join Al₂O₃ ceramics to metals, and the effect of Ti activity and reaction products between the Al₂O₃ ceramics and active brazing alloys on the joint strength have attracted the most attention in recent years. It has been proved^[4-6] that the reaction product at Ag-Cu-Ti/Al₂O₃ interface is relative to the Ti activity in the molten brazing alloy. It has been shown by Xin et al.^[4] and Ali Majed et al.^[5] that the microstructural evolution associated with the transformation of titanium oxide by prolonging the holding time at the brazing temperature. The interfacial chemistry in the AgCuTi-alumina system also

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Corresponding author: Li Ning, Ph. D., Professor, School of Manufacturing Science and Engineering, Sichuan University, Chengdu 610065, P. R. China, Tel: 0086-28-85405320, E-mail: lining@scu.edu.cn

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has been investigated by Voytovych et al. ^[6], and the results show that the reduction of the Ti-concentration in AgCu-based brazing alloy would cause the formation of sub-oxides of Ti such as Ti_2O_3 , and Ti_3O_5 . Zhu et al.^[7] investigated the interfacial microstructure and mechanical properties for Al₂O₃/Ag-Pd-Ti/ Kovar joints; their findings show that the type of titanium oxide is determined by the thickness of Ti layer, and the joint strength is influenced by the thickness of the reaction layer.

Recently it has been well accepted that during the active brazing process, Ti activity in the molten brazing alloy can influence the microstructures of the joins and then affect the mechanical properties of the brazed joints. It has also been reported that Ag can enhance Ti activity and Cu can suppress Ti activity in the molten brazing alloy^[8,9]; however, the relationship between Ti activity and Ti content in the brazing alloy is rarely mentioned because of the influence of other elements such as Cu and Ni^[10]. In order to identify the effect of Ti activity in the molten brazing alloy on the seam microstructure and the joint strength, in the present paper, Ag-Cu-Ti filler alloy is applied to braze Al₂O₃ and Fe-Ni-Co with different Ti contents under the reasons for the change of joint strength and the mechanisms for the formation of the brazed joints.

1 Experiment

The base materials are 95 wt% Al_2O_3 ceramic and Fe-33 wt%Ni-15 wt%Co alloy plates. The size of Al_2O_3 ceramic and Fe-Ni-Co alloy is shown in Fig.1. Fe-Ni-Co alloy plates with a thickness of 1 mm were treated with the surface nickel-plating to ensure a favorable spreadability of the brazing alloy on it. Generally, thickness of nickel coating on the Fe-Ni-Co alloy sheet was only ~6 μ m. The brazing alloy was a commercial Ag-Cu-Ti filler alloy (Lucas-Milhaupt, Inc) and the constituents of the Ag-Cu-Ti alloy is listed in Table 1. Their face for brazing were coated by Ag-Cu-Ti paste filler alloy using brushes and the thickness of the filler alloy was ~50 μ m (shown as Fig.1).

According to Fig. 1, Al₂O₃ ceramics and Fe-Ni-Co alloys are assembled after their surface for brazing was coated by commercial Ag-Cu-Ti filler alloys. The brazing process was performed in a metallic furnace consisting of molybdenum resistors located in a water-cooled stainless steel chamber,



Fig.1 Sketch of assembling jig for samples

1 a D C I = C I C I I C C I C C I C I D D D D C I C C C C	Table 1	Chemical	composition	of Ag-Cu-Ti	fillers alloy	(wt%)
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Fillers alloy	Ag	Cu	Ti
Ag-Cu-2Ti	-	34.09	1.73
Ag-Cu-4Ti	-	26.32	3.90
Ag-Cu-8Ti	-	23.92	7.89



Fig.2 Programming heating curve during brazing process

under a pressure of 3.0×10^{-3} Pa to avoid the oxidation of active Ti during heating. The thermal cycling process curve is shown in Fig.2 with a top brazing temperature of 890 °C. The tensile strength of brazed joints was tested by an electronic universal testing machine (RGX-M300) with a speed of 0.5 mm/min.

For the characterization of metallographic structure, the samples were ground into 10 mm with diamond discs along the direction perpendicular to the seam, and then the microstructure of the samples were observed by HITACHI S-4800 scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectrometer (EDS). The location for metallographic observation was marked by the black dash line in Fig.1. The different phases in the fracture surface were also identified by the X'Pert PRO X-ray diffractometer (XRD).

2 Results and Discussion

2.1 Mechanical property

The mechanical properties of the braze alloys with varied Ti content is illustrated in Fig.3. From Fig.3, the tensile strength of Al₂O₃/Ag-Cu-Ti/Fe-Ni-Co joints increases with increasing Ti content in the brazing alloys. It has been proved by Eustathopous^[6] that the increase of Ti content in the brazing alloy can cause a decrease in the contact angle, which indicate that the activity of Ti in the brazing alloy is influenced by the Ti content. It is clear that the increase of Ti content in the brazing alloy can improve the mechanical property of Al₂O₃/Ag-Cu-Ti/Fe-Ni-Co joint. It can thus be explained by the formation of an interface reaction layer adjacent to the Al₂O₃ ceramic ^[1-6].

2.2 Microstructures of Al₂O₃/AgCuTi/Fe-Ni-Co brazed joints

SEM microstructures of Al₂O₃/AgCuTi/Fe-Ni-Co joints are



Fig.3 Effect of the Ti content on the tensile strength of Al₂O₃/ Ag-Cu-Ti/Fe-Ni-Co joints

shown in Fig. 4. These images indicate that the joints are mainly composed of three parts: an interfacial reaction layer adjacent to Al₂O₃ ceramic (marked "A"), a brazing seam zone consisting of banded structures (marked "C"), lump phases (marked "D") and concave phases (marked "B"), and an interfacial reaction zone adjacent to Fe-Ni-Co alloy (marked "E"). These results reveal significant changes to the structure and morphology in the brazing zone as a function of Ti content.

Fig.4 shows a thick discontinuous interfacial reaction layer "E" between Fe-Ni-Co and AgCuTi brazing alloy, which indicates a violent interaction between Fe-Ni-Co and the molten brazing alloy. A faying surface between Ag-Cu-2Ti and Fe-Ni-Co is observed in Fig.4a and it is unassailable that the faving surface implies rare amount of the dissolution of Fe-Ni-Co to Ag-Cu-2Ti at a temperature of 890 °C for a holding time of 5 min. In comparison, an interfacial reaction zone with a tortuous joining line adjacent to Fe-Ni-Co alloy is also observed in Fig.4b and 4c. In addition, the reaction layer at Al₂O₃/AgCuTi interface is continuous and the thickness of reaction layer can be obtained by measuring the width of parallel dash lines shown in Fig.4. Comparing the three samples with varied Ti content in the brazing alloy, the thickness of reaction layer adjacent to Al₂O₃ ceramic increases with the rise of Ti content, and at a brazing temperature of 890 °C, it increases from 0.2~0.5 μ m to 1.4~1.7 μ m when the Ti content is

raised from 2 wt% to 4 wt% and it comes to 1.7 μ m with Ti content of 8 wt%, as shown in Fig. 4. It also should be noted that the sizes of banded structures become thicker and the amount of that increases as the Ti content increases.

The elemental distribution of the cross section of $Al_2O_3/AgCu4Ti/Fe-Ni-Co$ joint is presented in Fig. 5. These results show that Ti element is distributed to the Al_2O_3 ceramic surface (marked "A") and in the banded structure (marked "C"). Meanwhile, Ni element gives a similar distribution with Cu element in the interfacial reaction zone adjacent to Fe-Ni-Co alloy, but it also distributes to the Fe-Ni-Co surface due to the nickel-plating. It is should be noted that the Al and O is also distributed in the zone "A", and Cu element is distributed to the whole brazing seam.

In order to identify the formed phases in the brazing seam, the EDS point analysis was conducted to investigate the phase composition in each part as shown in Fig.4. Table 2~4 show the EDS point analysis of Ag-Cu-2Ti, Ag-Cu-4Ti and AgCu8Ti, respectively. The results of the phase composition in the brazing seam are also confirmed by XRD as shown in Fig.6. From these results, it can be concluded that zone A is the reaction layer, which has been reported elsewhere ^[1-5]. The composition of the reaction layer is mainly composed of Ti, Al and O.

In our previous work^[2] we have shown that the lump phases (marked "D") are Cu-rich and the concave phases (marked "B") are Ag-rich. Similarly, the (Cu, Ni) solid solutions (marked "E") also form adjacent to Fe-Co-Ni alloy, because lots of Ni from Fe-Co-Ni meet partial Cu-rich liquid from the molten brazing alloy of AgCuTi^[4]. The EDS results show that the composition in zone A, C is varied with different brazing alloys. "A" zone is composed of Ti, O and Al; it can be inferred that Ti-Al intermetallic and titanium oxide formed at Al2O3/AgCuTi interface due to the diffusion of active element Ti^[11]. The EDS point analysis indicates that the variety of Ti-Al intermetallic and titanium oxide is dependent on Ti content in the brazing alloy. According to the EDS point results and the XRD patterns in the AgCu2Ti brazing seam, zone "A" consists of TiO2 and Ti₉Al₂₃; as the Ti content increases, the phase composition in zone "A" turns into Ti₃O₅ and TiAl with Ag-Cu-4Ti brazing alloy and then turns into TiO and Ti₃Al with Ag-Cu-8Ti brazing



Fig.4 SEM micrographs of Al₂O₃/AgCuTi/Fe-Ni-Co joints for Ag-Cu-2Ti (a), Ag-Cu-4Ti (b), and Ag-Cu-8Ti (c) at a brazing temperature of 890 °C



Fig.5 EDS elements Ti-Ag-Al-Ni-Cu-O distribution of the cross-section of Al₂O₃/Ag-Cu-4Ti/Fe-Ni-Co brazing joints in the box zone of Fig.4b



Fig.6 XRD patterns from the fracture surface of Al₂O₃/Fe-Ni-Co joints with the brazing alloys of Ag-Cu-2Ti (a), Ag-Cu-4Ti (b), and Ag-Cu-8Ti (c) from the side of Fe-Ni-Co

alloy. The EDS results also show that the phase composition in zone "C" is also changed as the Ti content increases. The EDS point results in Table 2 show that zone "C" is mainly composed of Ni, Cu, Ti. The phase composition in zone "C" can be predicted that the zone "C" in AgCu₂Ti brazing seam mainly consist of CuTi₂, NiTi₂ and Ag-rich and zone "C" in Ag-Cu-4Ti brazing seam mainly consist of Cu₃Ti, NiTi and Ag-rich; otherwise, as the Ti content is up to 8 wt% in the brazing seam, the zone "C" mainly comprise Ni₄Ti₃ phase and Ag-rich. The typical microstructure of the brazing joint of Ag-Cu-4Ti is Al₂O₃/TiAl+Ti₃O₅/NiTi+Cu₃Ti+Ag(s,s)/Ag(s,s)+ Cu(s,s)+(Cu,Ni)/Fe-Ni-Co.

2.3 Microstructure analysis

The similar microstructures of brazed joints were obtained with different Ti content brazing alloy, However, the microstructures of the brazing zone are slightly different, which depend on the Ti content in the brazing alloy. When Ag-Cu-2Ti was used to braze Al₂O₃/Fe-Ni-Co, the thickness of the interfacial reaction layer "A" was thinner, which indicates a weak interaction between Al_2O_3 and the brazing alloy. Therefore, it can be predicted that the activity is weaker. When Ag-Cu-4Ti was used to braze Al_2O_3 /Fe-Ni-Co, the thickness of the interfacial reaction layer "A" became thicker and the interaction between Ti and Al_2O_3 ceramic became stronger, which indicates the activity of Ti in the active brazing alloy was enhanced. However, with 8 wt% Ti in the brazing alloy, the thickness of the interfacial reaction layer "A" increases, but as compared to Ag-Cu-4Ti, the increase amplitude is smaller.

During the brazing process, the microstructure and composition in the reaction zone are determined by the metallurgical reaction between the molten brazing alloy of Ag-Cu-Ti and the base materials of $Al_2O_3^{[12]}$. Under these conditions, the molten brazing alloy can be regarded as regular solutions and the interaction between the molten brazing alloy and Al_2O_3 are essential for the brazing joints^[13]. From the view of thermodynamics of alloys^[14], the relationship between the molten brazing

Table 2EDS point analysis of Ag-Cu-2Ti in Fig.4a (at%)

		1			8		0 (,
Zones	0	Al	Ti	Fe	Co	Ni	Cu	Ag
А	28.0	3.6	7.1	1.0	0.5	16.9	3.8	39.1
В	24.8	3.6	0.7	0.6	0.1	1.4	9.1	59.7
С	7.0	2.7	8.5	1.0	0.4	21.6	56.9	1.9
D	4.1	2.6	1.7	-	-	6.8	83.4	1.4
Е	8.6	3.7	-	5.7	1.3	25.6	52.5	2.6

Zones	0	Al	Ti	Fe	Co	Ni	Cu	Ag
А	30.8	9.3	19.8	8.9	2.6	24.5	3.4	0.7
В	2.4	4.6	3.9	-	-	1.9	17.5	69.7
С	7.4	1.9	16.4	2.5	3.0	38.7	16.8	13.3
D	2.5	1.9	4.6	1.0	0.4	15.1	72.9	1.6
Е	5.8	2.1	0.4	4.4	1.8	55.1	29.2	1.2

Table 4EDS point analysis of Ag-Cu-8Ti in Fig.4c (at%)

	1			0		0	· · ·	
Zones	0	Al	Ti	Fe	Co	Ni	Cu	Ag
А	25.8	23.5	8.2	0.3	0.2	22.2	17.8	2.0
В	0.7	0.1	0.2	-	0.6	2.7	8.7	87.0
С	1.0	0.5	16.7	0.5	0.2	49.4	10.6	21.1
D	0.8	0.6	1.7	-	-	9.8	85.7	1.4
Е	0.7	0.2	1.4	4.5	1.8	24.9	66.2	0.3

alloy of Ag-Cu-Ti and the activity of Ti were analyzed. As for the liquid Ag-Cu-Ti ternary regular solutions, the relation between thermodynamic activity a_{Ti} and mole fraction of each composition in this solutions can be expressed as^[14]:

 $RT \ln a_{Ti} = (1-x_{Ti})^2 L_{AgTi} + RT \ln x_{Ti} + (x_{Cu})^2 L_{AgCu} + x_{Cu}(1-x_{Ti}) W_{CuTi}$ (1) where x_{Ti} and x_{Cu} are mole fraction of Ti and Cu, respectively; Ris gas constant; T is thermodynamic temperature; L_{AgTi} and L_{AgCu} are the binary group interaction energy in Ag-Ti solutions and the binary group interaction energy in Cu-Ti solutions; W_{CuTi} is the Cu, Ti interaction energy in Ag-Cu-Ti ternary solutions. According to Ref.[15,16], the thermodynamic activity a_{Ti} in the brazing alloy can be expressed as:

 $RT \ln a_{\text{Ti}} = 32.83(1 - x_{\text{Ti}})^2 + 15.50x_{\text{cu}}^2 - 16.14x_{\text{Cu}}(1 - x_{\text{Ti}}) + RT \ln x_{\text{Ti}} (2)$

The activity of Ti in the brazing alloy directly affects the interface reaction between ceramic and filler alloy, and then affects the microstructure of the interface. From Eq. (2), it can be concluded that the activity of titanium is affected by the content of titanium and copper; therefore the effect of titanium and copper content on the microstructure of the solder is discussed.

When the titanium content is lower (Ag-Cu-2Ti), the activity of titanium is relatively weak and the reaction between the brazing alloy and the ceramic is not sufficient or even difficult to carry on ^[10]. Moreover, because the interaction energy between Cu and Ti is -16.14 kJ/mol ^[16], there is a great affinity between copper and titanium to form intermetallic compounds. Consequently, a certain amount of Ti-Cu intermetallic compounds such as CuTi₂ are formed in the brazing zone. In this case, copper can also decrease the activity of titanium in the molten brazing alloy due to the interaction between Ti and Cu. Therefore, only a small quantity of titanium is diffused to the interface of Ag-Cu-Ti/Al₂O₃, resulting in the formation of TiO_2 and Ti_9Al_{23} in the reaction layer and a thinner or incomplete reaction layer is also observed in the zone "A" by SEM.

The increase of Ti content and decrease of Cu content in the brazing alloy, such as Ag-Cu-4Ti, can increase the activity of Ti to 0.939 listed in Table 5. Under this conditions, much more Ti atoms diffuse to the interface between the brazing alloy and Al₂O₃ ceramic, which may decrease the reaction severity of Ti-Cu; therefore, Ti-Cu intermetallic compounds such as Cu₃Ti are formed in the brazing zone and the thickness of the reaction layer increases compared to that of Ag-Cu-2Ti. Consequently, the phase composition in the reaction layer is transformed to Ti₃O₅ and TiAl. With the addition of 8 wt% Ti in the Ag-Cu based brazing alloy (Ag-Cu-8Ti), the thermodynamic activity of Ti in the alloy is raised to ~1 listed in Table 5, and therefore, the thickness of reaction layer "A" increases to a maximum value. On this occasion, the reaction layer "A" is mainly composed of TiO and Ti₃Al. From these results, it can be concluded that the reaction product in the reaction layer is determined by the activity in the molten brazing alloy, which is dependent on the brazing temperature and the content of Ti and Cu.

It should be noted that nickel atoms also diffuse into copper layer at the side of Fe-Ni-Co alloy, which leads to the formation of Cu-Ni solid solution ^[17], and the interface migration is unidirectional. Thus the interfacial reaction zone "E", consisting of Cu-Ni solid solution, forms at the side of Fe-Ni-Co alloy, and then the Ni atoms continue to diffuse into the molten brazing alloy and react with Ti drastically to form Ni-Ti intermetallic compound such as NiTi₂, NiTi and Ni₄Ti₃. Certainly, Ti also reacts with Cu to form Cu-Ti compound. Nevertheless, with addition of 8 wt% Ti in the brazing alloy, there is little Ti-Cu intermetallic compound in the brazing joints which is not detected by XRD and the reaction between Ti and Ni would be aggravated, and some Ni-Ti intermetallic compounds such as Ni₄Ti₃ form.

2.4 Fracture analysis

When the reaction layer is not complete such as Ag-Cu-2Ti, the tensile strength is lower and fracture always occurs at the discontinuous reaction layer, which could not alleviate the larger residual stress between ceramic and metal generated during the cooling process.

When the reaction layer is thick enough such as Ag-Cu-4Ti or Ag-Cu-8Ti, the tensile strength is higher because some transitional oxides such as TiO can alleviate the larger residual stress. Under these conditions, the tensile fracture is often occurs in the reaction layer "A", resulting from the intensity

Table 5 Ti activity in the molten brazing alloy Ag-Cu-Ti

Filler alloy	$x_{\rm cu}/{\rm at}\%$	$x_{Ti}/at\%$	a_{Ti}
Ag-Cu-2Ti	38.97	3.44	0.567
Ag-Cu-4Ti	36.28	7.13	0.939
Ag-Cu-8Ti	34.41	13.88	1

of local stress around Ti-Al intermetallic compounds, which is hard and brittle^[18], and then the micro-cracks appear because of the existing of stress between Ti-Al intermetallic compounds and titanium oxide due to the differences in elastic-plastic^[2].

3 Conclusions

1) Good reaction layer is formed by metallurgical reaction at the interface of Al_2O_3/Ag -Cu-4Ti, and the typical microstructure is $Al_2O_3/TiAl+Ti_3O_5/NiTi+Cu_3Ti+Ag(s,s)/Ag(s,s)+$ Cu(s,s)+(Cu,Ni)/Fe-Ni-Co brazed at 890 °C for 5 min. Compared with Ag-Cu-4Ti filler alloy, the microstructure changes slightly when the addition of Ti is up to 8 wt% in Ag-Cu-Ti filler except for the increase of the thickness of reaction layer adjacent to Al_2O_3 ceramic.

2) The activity of active elements Ti in the brazing alloy directly affects the interface reaction between ceramic and filler metal, and then affects the microstructure of the interface. The activity of Ti is influenced by the content of titanium and copper in the brazing alloy.

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Ti 活度对 Al₂O₃/Ag-Cu-Ti/Fe-Ni-Co 钎焊接头力学性能和微观组织结构的影响

辛成来,李 宁,颜家振,曹永同 (四川大学,四川 成都 610065)

摘 要:采用 AgCuTi 钎料实现了 Al₂O₃ 陶瓷与 Fe-Co-Ni 合金的钎焊连接,并调查了不同钛含量的钎料对 Al₂O₃/Ag-Cu-Ti/Fe-Ni-Co 钎焊 接头机械性能和微观组织结构的影响。利用扫描电镜(SEM),X 射线能量谱仪(EDS),X 射线衍射仪(XRD)及电子万能试验机研究 了钎焊接头的力学性能和微观组织结构。结果表明,钛含量的增加明显提高 Ag-Cu-Ti 钎料与 Al₂O₃ 陶瓷的相互作用,在 Al₂O₃/Ag-Cu-Ti 界面生成一层由 Ti-Al 和 Ti-O 化合物组成的反应层。Al₂O₃/Ag-Cu-Ti/Fe-Ni-Co 钎焊接头的抗拉强度随钛含量的增加而增加,当钛含量 提高到 8%(质量分数)时,抗拉强度达到最大值 78 MPa。通过微观组织结构分析发现,采用 AgCu4Ti 在 890 ℃保温 5 min 的条件下 可以获得较好的钎焊接头,典型接头的微观组织结构为 Al₂O₃/TiAl+Ti₃O₅/NiTi+Cu₃Ti+Ag(s,s)/Ag(s,s)+Cu(s,s)+(Cu,Ni)/Fe-Ni-Co。采用 Ag-Cu-8Ti 获得的钎焊接头的界面反应层与 Ag-Cu-4Ti 差异不大,但反应层稍微增厚,并伴有 TiO 和 Ti₃Al 在 Al₂O₃/Ag-Cu-Ti 界面生成。 关键词:氧化铝陶瓷; Fe-Co-Ni 合金; 钛活度; 机械性能; 微观结构

作者简介: 辛成来, 男, 1985 年生, 博士, 四川大学制造科学与工程学院, 四川 成都 610065, 电话: 028-85405320, E-mail: 523742033@qq.com