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Brazeability of Ni-Sn-Ti Filler Metals against Si_3N_4

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Abstract

A series of Ni-20Sn-xTi alloys, where x changed from 0 to 15 at%, has been developed for brazing Si_3N_4 in vacuum. The wettability and bonding strength of the alloys were evaluated by sessile drop and shear testing methods. The contact angle of the alloys on Si_3N_4 shows the lowest at 10 at% Ti in the alloys, and the tendency of strength of 4 joints brazed with Ti content in the alloys is coincident with tendency of the contact angle of the alloys. The Ni-20Sn-10Ti alloy, which processes the lowest contact angle, yields the highest bonding strength against Si_3N_4 In other words, the alloy which possesses the high wettability against the ceramics, shows the good bonding strength against the ceramics.

KEY WORDS: (Ni filler metals) (Ni) (Sn) (Ti) (Contact angle) (Wettability) (Brazing) (Bonding) (Ceramic joining)

1. Introduction

Joining of ceramics to metals is a fundamental technology for expanding ceramics application, because metals compensate for the brittleness of ceramics. Silver base and copper base filler metals containing titanium have been used for joining Si_3N_4 [1,2,3]. Tamai and Naka have reported that Ti in the filler metals reacts preferably with ceramics and promotes strong bonding [4]. Nishimoto et al.[5] have also investigated the strength of the brazed Si_3N_4 with Ti containing Cu base filler metals. Urai et al reported that the addition of Sn to Cu-Ti filler metals reduced the melting points, and improved the brazeability of the Cu-Ti filler metals [6].

Nickel base alloys instead of the silver and copper base alloys are also used for brazing heat resistant alloys [7], [8]. This work also tries to develop nickel base alloys containing Sn for brazing Si_3N_4, and investigates the brazeability of the nickel base alloys.

2. Experimental Procedure

Presureless sintered Si_3N_4 containing a few percent alumina as a sintering aid was used. A series of Ni-20Sn-xTi (x=0,5,10,15) alloys, whose compositions are listed in Table1 where the number designates atomic percent of Si_3N_4 at 6 mm diameter and 3 mm thickness to Si_3N_4 at 15 mm diameter and 3 mm thickness using the Ni-20Sn-xTi filler metals was done at high temperatures in vacuum. The size of the filler metal was 6 mm diameter and 0.1 mm thickness.

The melting points of the alloys were measured by differential diameter with a heating rate of 20K/min. The strength of the brazed Si_3N_4 joints was evaluated by fracture shear testing with a crosshead speed of 1 mm/min.

The wettability of the alloys was evaluated using a sessile drop metal in Fig.1. The contact angle of a sessile drop of the alloy on Si_3N_4 in vacuum was measured using a camera as shown in Fig.2. The microstructure of the sessile drop and the joint were investigated by using an electron probe microanalyser.

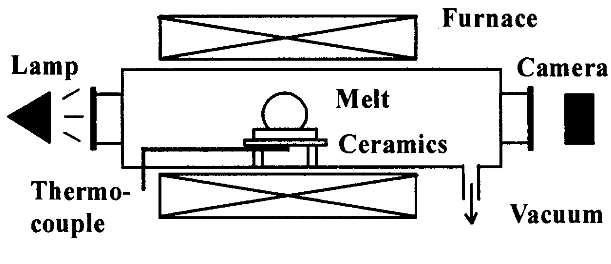

Fig. 1 Sessile drop technique apparatus.

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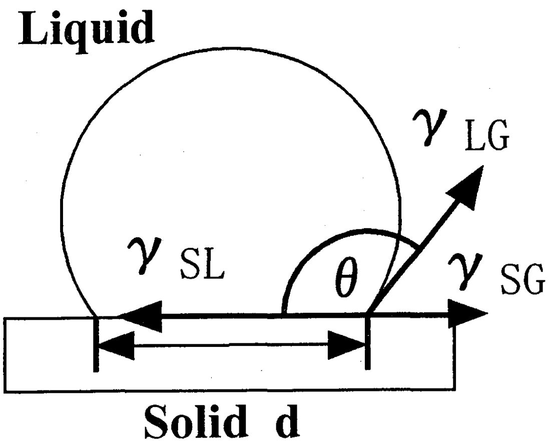

Fig. 2 Schematic of contact angle of liquid drop.

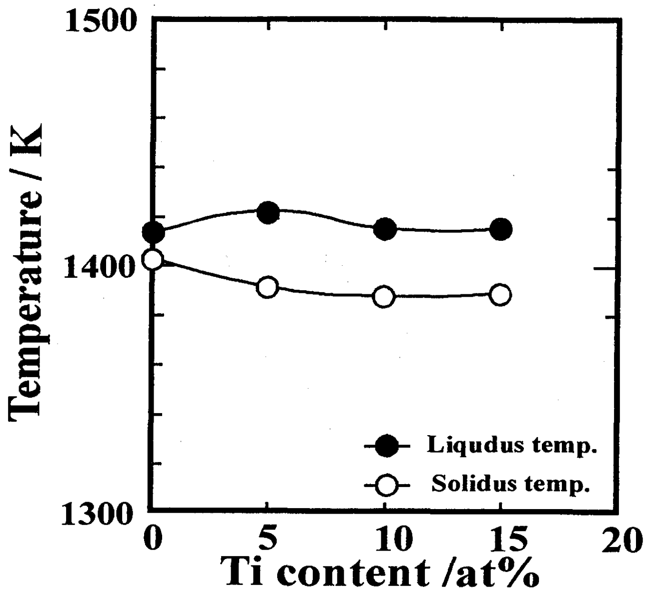

Fig. 4 Change in the solidus and liquidus temperatures of Ni-20Sn-xTi alloys with Ti content.

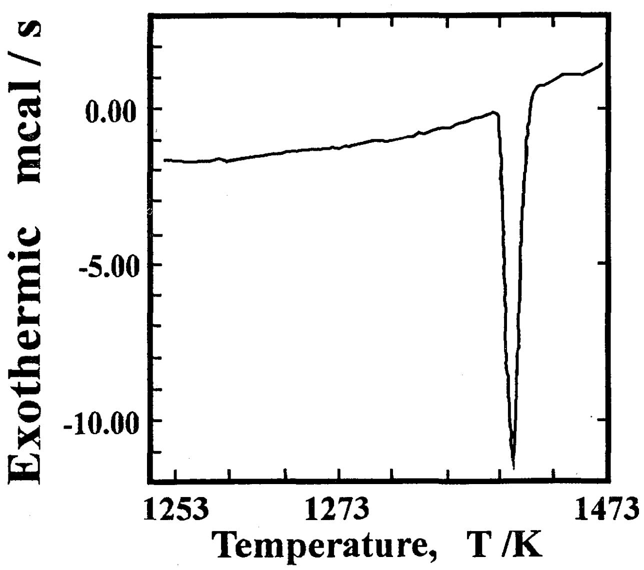

Fig. 3 DSC curve of Ni20Sn10Ti alloy.

3. Results and Discussion

Melting points of Ni-20Sn-xTi alloys with a constant Sn content of 20 at% were measured by measuring the DSC curve. The example of the DSC curve of Ni-20Sn-10Ti alloy is shown in Fig.3. The alloy shows the endothermic peak, and the solidus and liquidus temperatures are 1389 K and 1416 K, respectively. Fig.4 represents the change in the solidus and liquidus temperatures of Ni-20Sn alloys as a function of Ti content. The addition of Ti to Ni-20 at% Sn alloy lowers the solidus and liquidus temperatures. The solid and liquid temperatures of Ni-20Sn-10Ti alloy show the lowest values of 1389 K and 1416, respectively.

The wettability of Ni-20Sn-xTi alloys was evaluated by measuring the contact angle on Si3N4. Fig.5 and 6 show the change in contact angle of Ni-20Sn-xTi alloys with holding time at 1473 K. Ni-20Sn alloy yields the constant angle of 150 degree. and does not wet Si_3N_4.

The addition of Ti lowers the contact angle of the molten Ni-20Sn alloys. The degree of decrease in the contact angle depends on the Ti content in the alloys as shown in Fig.5. Ni-20Sn-7.5Ti alloy quickly lowers the equilibrium at the initial time, and reaches 112 degree at 4.5 ks. Ni-20Sn-10Ti alloy reaches gradually the equilibrium angle of 98 degree, and the value of the alloy is the lowest among alloys as shown in Fig.6. The further addition of Ti content over 10 at% increases the

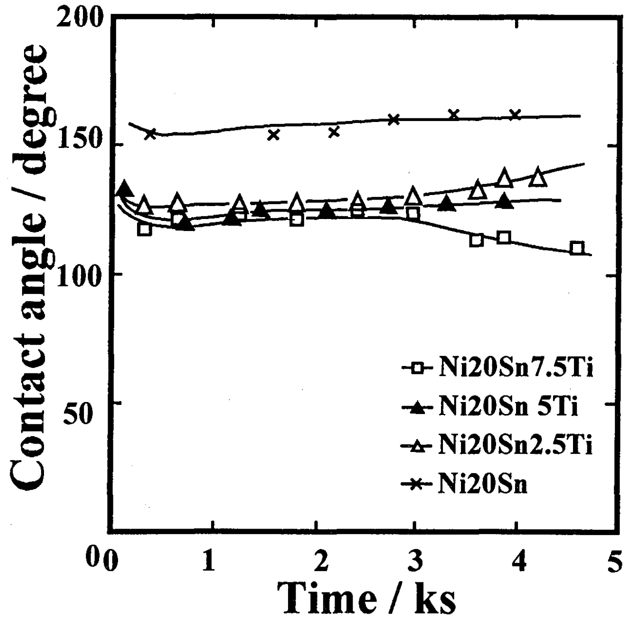

Fig. 5 Change in contact amble of Ni-20Sn-xTi alloys with holding time at 1473 K.

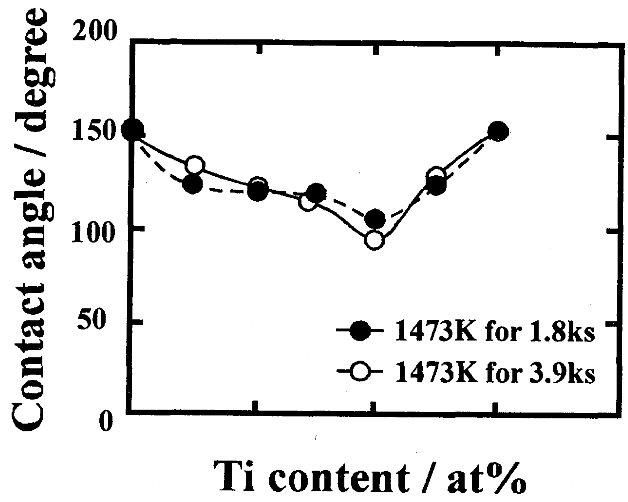

Fig. 7 Ti content dependence of the contact angle at 1473 k for 1.8 ks and 3.9 ks.

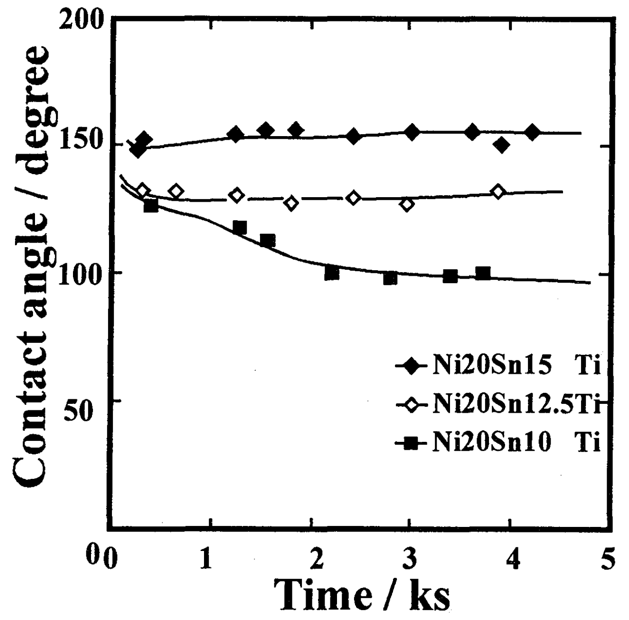

Fig. 6 Change in contact amble of Ni-20Sn-xTi alloys with holding time at 1473 K.

contact angle. Ni-20Sn-15Ti alloy with no change of contact angle with time shows the higher value of 155 degree. Ni-20Sn-10Ti alloy yields the lowest contact angle among the alloys. The Ti content dependencies of the contact value of the alloys at 1473 K for 1.8ks and for 3.9 ks is represented in Fig.7. In the figure the contact angles at 1.8 ks and 3.6 ks were plotted against Ti content in the alloys. Those tendencies of the contact angles against Ti content are almost the same. The contact angle of Ni-20Sn alloy lowers with adding Ti, and reaches the lowest at 10 at% Ti, and rises with further adding Ti. The addition of Ti to Ni-20Sn alloy effectively lowers the contact angle. This implies that Ni-20Sn-10Ti alloy shows a good wettability against Si_3N_4, and the excess addition of titanium over 10 at% degrades the wettability of Ni-20Sn alloy.

Fig.8 shows the microstructure of a sessile drop cooled down to room temperature for Ni-20Sn-10Ti alloy on Si_3N_4. Ti dissolved in the nickel phases in large needle like primary phase and eutectic phases. The composition of the primary nickel phases (part 1 in Fig.8) is 8.8 at% Sn, and 16.4 at% Ti and balance of Ni. The eutectic phases (part 2) in Fig.8 are composed of white Ni_3Sn_2 and grey Ni. After cooling the sessile drop adhered to the Si_3N_4. The bonding of the sessile drop is due to the nickel phases adjacent to Si_3N_4 which adhere to the ceramic, though titanium in the Ni-20Sn-10Ti alloy doesn't react with the ceramic. The direct bonding of nickel has been also reported [8].

Since the Ni-20Sn-10Ti alloy shows the contact angle of 98 degree and adheres after cooling down, the

Fig. 8 Microstructure of a sessile drop cooled down to room temperature for Ni-20Sn-10Ti alloy on Si_3N_4.

alloy could be applicable as a filler metal for brazing Si_3N_4. After brazing Si_3N_4 to Si_3N_4 using a series of Ni-20Sn-xTi filler metals, the strength of the Si_3N_4 joints were measured by fracture shear testing. The change of the joints are shown in Fig.9. The Ni-20Sn without Ti doesn't give the bonding , and the strength of the Si_3N_4 joints increases to 111Mpa with Ti content up to 10 at% Ti in the Ni-20Sn alloys. The excess addition of Ti content over 10 at% to the Ni-20Sn alloy degrades the strength of the joints. This decrease of the strength of the joints is in agreement with the decrease in the contact angle with the excess Ti content. The reactivity of the alloys degrades with the excess addition of Ti over 10 at%.

Fig.10 represents the micostructure of Si_3N_4 joint brazed with Ni-20Sn-10Ti alloy at 1473 K for 1.8ks. The characteristics of the microstructure of the Si_3N_4 joint (part 1,2) are coincident with that of the sessile drop of the Ni-20Sn-10Ti alloy on Si_3N_4. The nickel phases adjacent the Si_3N_4 of the joint form the strong bonding with the ceramic after cooling down. The tendency of the strength of the Si_3N_4 brazed joint against Ti content agrees with the tendency of the contact angle of the alloys. The Ni-20Sn-10Ti alloy with the good wettability yields a strong bond with the Si_3N_4.

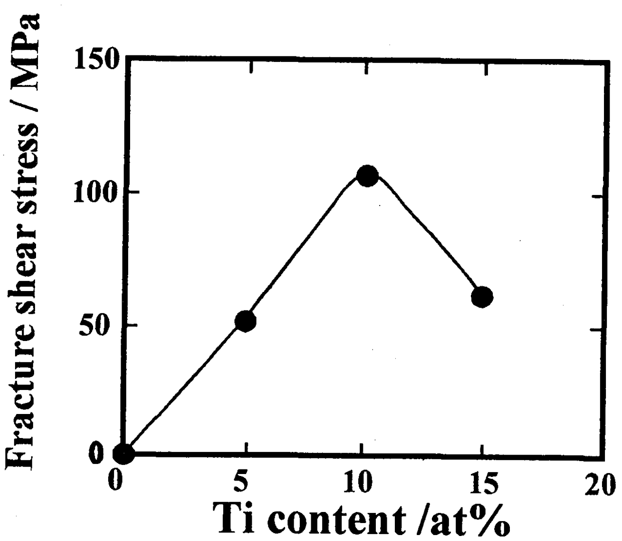

Fig. 9 Change of strength of Si_3N_4/Si_3N_4 joints with Ti content in filler metals.

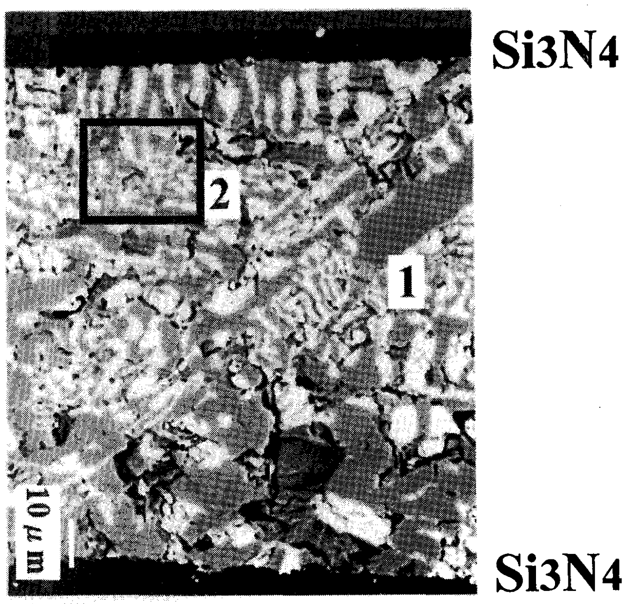

Fig. 10 Microstructure of Si_3N_4/Si_3N_4 joint brazed with Ni-20Sn-10Ti allloy at 1473K for 1.8 ks.

4. Conclusions

A series of Ni-20Sn-xTi filler alloys has been developed for brazing Si_3N_4 in vacuum. The wettability and the bonding strength of the alloys against Si3N4 were evaluated by measuring the contact angles in sessile drops of the alloys, and fracture shear strength of Si_3N_4 brazed joint with the alloys in a shear testing. The Ni-20Sn-10Ti alloys show the lowest contact angle of 98 degree at 1473K for 3.9ks. The excess content of Ti in the alloy yields a higher value, and degrades the wettability of the alloys. The tendency of the contact angle of the alloys against Si_3N_4 is incident with the bonding strength of the alloys against the ceramic. The strength of the Si_3N_4 joint brazed with Ni-20Sn-10Ti alloy shows the maximum value of 111 MPa, and the strength of joint degrades with the excess content of Ti over 10 at%.

The good wettability and bonding of Ni-20Sn-10Ti alloy against Si_3N_4 comes from the bonding of nickel in the alloys to the ceramic.

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