Microstructural Analysis of Porous Nickel Brazed to Copper and Stainless Steel using Different Brazing Filler Metals

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ABSTRACT

The microstructures of brazing porous Nickel (Ni) to copper and stainless steel (Cu/Porous Ni/SS304) was investigated. A porous Ni with pore densities of 15 PPI (pores per inch) and filler with compositions of 72Ag-28Cu and 77.4Cu-9.3Sn-7P-6.3Ni (Ag: Silver; Cu: Copper; Sn: Tin; P: Phosphorus; Ni: Nickel) were employed. The brazing process was conducted at different brazing temperatures: 830˚C, 870˚C and 910˚C for 15 minutes brazing time with heating and cooling rate of 10˚C/min, respectively. The aim of this research is to analyse and compare the microstructure of using different types of filler metal in order for joint porous Ni to copper and stainless steel. According to the initial observations from an optical microscope (OM) after cross-section, the used of filler metal BAg-8 and VZ2250 acquired a bonding joining than the used of filler metal BAg-8 solely. Thus, Field Emission Scanning Electron Microscope (FESEM) equipped with Energy Dispersive X-Ray Spectroscopy (EDS) was used to characterise the bonding of microstructure of filler metal BAg-8 and VZ2250. Besides, the joint strength of Cu/Porous Ni/SS304 with filler metal BAg-8 and VZ2250 was evaluated with shear strength at different brazing parameters. Consequently, it can be concluded that used of filler metal BAg-8 and VZ2250 performed a better joint microstructure as compared to the used solely of filler metal BAg-8.

Keywords: Brazing, Copper, Stainless Steel, Porous Ni, Microstructures

1 Introduction

Porous metal plays a significant role in thermal application such as heat exchanger, filtering, heat sinks, petrochemical industry, aircraft, and automotive systems [1-4]. Porous metal is an integral part of cooling system due to its higher surface area and lower product cost where heat can be transferred coefficiently [1, 5, 6]. Previous research on porous metal heat exchangers (HEs) examined the advantages of their different types and porosities [7-10].

In general, there are two (2) types of porous metals: open-cell and closed cell. The voids in open-cell porous metal are interconnected which provides pathways for fluid flow. The voids in closed-cell porous metal, on the other hand, are not connected and are separated by solid metal matrix. As for a HEs application, open-cell porous metal have proven to be highly suitable due its superior thermos dynamics properties and permeability for fluid flow [7]. Besides, the use of open-cell porous metal as compact HEs tends to increase the thermal resistance [10]. This statement has been tested experimentally and is considered as an alternative for compact HEs material to replace conventional fins [9, 11].

Apart from that, the heat transfer performance of high porosity porous metal has been investigated by Bin Liu and the team. The study showed that 90% of porosities has the best performance in comprehensive

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heat transfer coefficient [12]. Therefore, the effective thermal conductivity of the porous metals strongly depends on porosity.

However, the fabrication of porous metal in HEs is challenging since it must be developed by contacting porous metals with base metals. Among all of the methods available for different metal joining, brazing may be the most versatile and an effective way to join porous metal and base metals even up to high temperature (max: 1200˚C) [13-15]. Nevertheless, there are limited studies on brazing of porous metal applied for heat exchangers have been reported in the literature [14, 15].

Furthermore, one of the most useful engineering materials is copper and copper alloys. However, due to interesting copper-based material properties such as good strength, corrosion resistance, conductivity, and ductility would make copper become more suitable for a comprehensive variety of applications. Shiri and the team have discovered that stainless steel has a low thermal conductivity in comparison to copper and its alloys. Due to the limited thermal conductivity of these alloys, heat is not dissipated to the environment when they are utilised at high temperatures. Therefore, the heat dissipation during high temperature applications and the likelihood of deleterious phases forming lowered by joining copper and stainless steel [16].

For that reason, it is important to investigate the microstructure for prediction to identify the suitable filler metals for brazing porous Ni to copper (Cu) and stainless steel (SS304) as heat exchangers application. Porous Ni is chosen due to its advantages in corrosion resistance [17]. Apart from that, inadequate research on brazing with porous Ni as compared to porous Cu has become the urgency of this study.

In the preliminary study, Ag-based brazing filler metal was applied for both sides to join porous Ni to Cu and stainless steel. However, at the stage of analysis, it was observed that the microstructure is better at the SS304 interface only and not at both interfaces (Cu and SS304). Therefore, the current study improved the selection of brazing filler metal by replacing amorphous Cu-based to be applied at one side of the Cu interface while remaining the SS304 interface with Ag-based filler metal. Thus, this paper discussed the microstructure for both conditions of the different brazing filler metals with shear strength data.

2 Experimental Details

In this research, pure solid copper (Cu) and stainless steel (SS304) were used as a base metal with dimension of 20 x 20 x 3 mm^3, respectively. On the other hand, 24 mm thick of 15 PPI (pores per inch) porous Ni (supplied from Japan) with porosity of 96.14% were cut using EDM wire cut machine into 10 x 10 mm dimensions. Two (2) types of brazing filler metal with compositions of 72Ag-28Cu (wt. %) known as BAg-8 and 77.4Cu-7Ni-9.3Sn-6.3P (wt. %), also known as VZ2250 (VACUUMSCHMELZE GmbH & Co. Kg. Hanau, Germany) were used in this study as comparison. Both filler metal with thickness of 30 µm was prepared in approximated size of 18 x 18 mm^2. Since there are two (2) types of filler metal used in this experiment, Table 1 presented the sample Id# for each brazing parameters.

All of the materials were arranged in the form of sandwich configuration before brazing, as shown in Figure 1 (a) (I and II). The brazing process were conducted in a high vacuum furnace, Model Mini-Vac-II (10^{-5} Torr) (Tokyo Vacuum Co., Ltd.) with brazing temperature 830 ˚C, 870 ˚C and 910 ˚C for 15 minutes brazing time. The heating and cooling rate are approximated 10 ˚C/min, respectively in order to ensure the brazing filler metal is fully melt and enough to provide a good bonding to join Cu/Porous Ni/SS304.

After brazing, the samples were left for cooled in the vacuum furnace. Prior to characterization process, the samples were ground using abrasive paper (500, 600 and 800 grit) and polished until mirror finished with Alumina powder (0.05 µm). Then, the Optical Microscope (OM) (Model: Olympus BX61, Japan) was used to identify the joint bonding after standard metallographic techniques. Then, Field Emission Scanning Electron Microscope (FESEM) was used to captures the bonding images. Next, in order to determine the specimens' content, Energy-Dispersive X-Ray Spectroscope (EDS) equipped with elemental analysis software was used. Finally, shear test was conducted to evaluate the bonding strength. Figure 1 (b) depicts the illustration of shear test using a Universal Testing Machine (Model: Instron 3369) equipped with Bluehill 2.0 software and fixed with a 50 kN load working in displacement control mode at 1 mm/min speed. Before the testing begin, both the force and gauge length were reset to zero. The measuring device on the load automatically recorded and converted the data into assessable data. To guarantee the validity of the data, each specimen was tested with three samples.

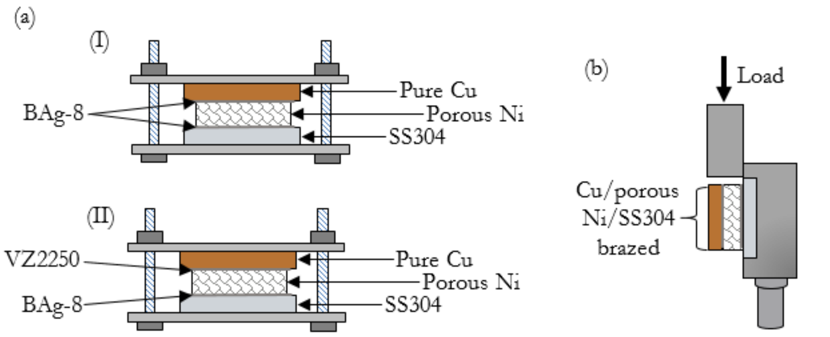

Figure 1: (a) *(I and II)) Sample arrangement in a specific clamp*. **(b)** *Shear*

Table 1: *Sample Id# for each brazing parameters used in this research.*

Brazing Parameters	Sample Id #
830˚C - Cu/BAg-8/Porous Ni/BAg-8/SS304	A1
830˚C - Cu/VZ2250/Porous Ni/ BAg-8/SS304	A2
870˚C - Cu/BAg-8/Porous Ni/BAg-8/SS304	B1
870˚C - Cu/VZ2250/Porous Ni/ BAg-8/SS304	B2
910˚C - Cu/BAg-8/Porous Ni/BAg-8/SS304	C1
910˚C - Cu/VZ2250/Porous Ni/ BAg-8/SS304	C2

3 Results and Discussion

3.1 Microstructural Characterization

The brazing process was successfully conducted using the parameters described in the methodology. By naked eyes, it can be seen the porous Ni was joined on both sides for all filler metals employed in this study. Furthermore, the structure of porous Ni still maintains its original size even after brazing with a slight change in its colour from grey to white. The cross-section of the brazed specimens was analysed using an OM and FESEM with EDS point analysis to examine the distributions of the elements. Figure 2 shows the microstructure of all samples brazed at 15 minutes. For each of the samples, 2 interfaces were denoted, Interface 1: The interface of Cu/Porous Ni and Interface 2: The interface of Porous Ni/SS304. It can be observed that the filler metal diffused on both sides of the base metals. The diffusion of filler metal can be identified by different pattern in white and grey colour in the FESEM images below.

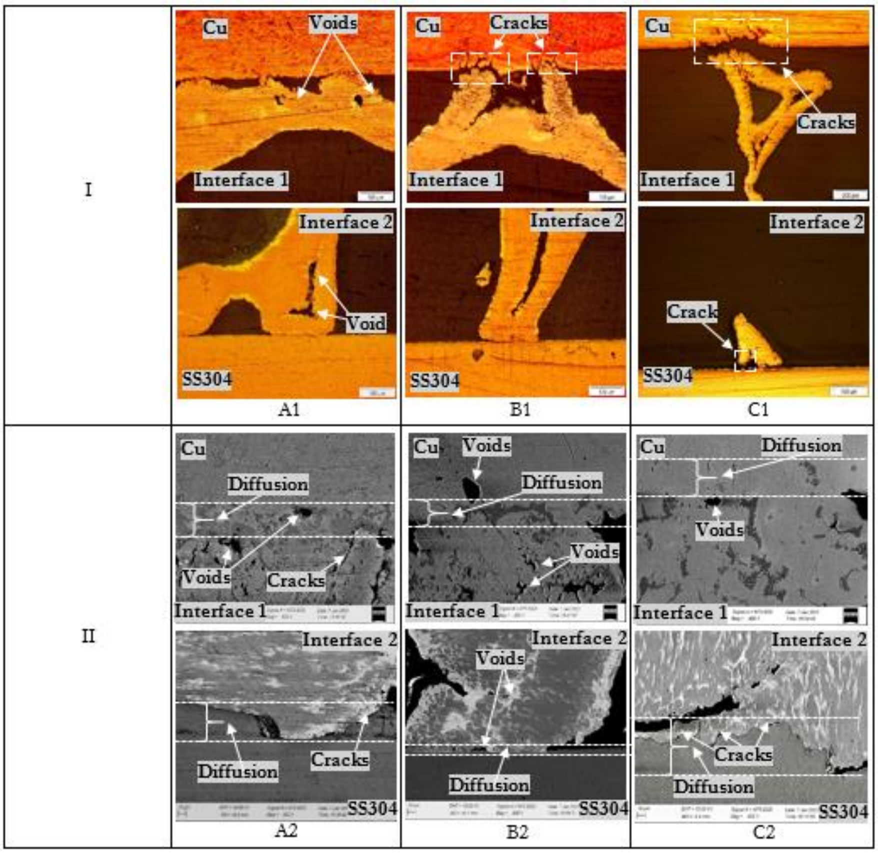

Figure 2: (I) *OM images and* **(II)** *FESEM images for the microstructure of all samples brazing at 15 minutes for both Interface 1 and 2.*

Figure 2 (I) (A1, B1 and C1) displays the microstructure of filler BAg-8 for both sides. As the brazing temperature increases, the joint microstructure significantly cracks. It can be seen in Figure 2 (B1 and C1) as the filler metal could not effectively wet the Cu interface. This might happen due to the element of either Ag or Cu in the eutectic that decreased the brazing wetting. Besides, reaction of the filler metal (BAg-8) with Ni that mainly originated from porous Ni during brazing could be attributed to the formation of cracks and eroded on the microstructure. The wettability of BAg-8 with Ni is limited as Ni is difficult to soluble with Ag as compare to Cu [18]. This statement can be proven in the EDS analysis which will be discussed later that reveal element Ni and Ag at both interfaces for all brazing parameters are the main elements contributing to the formation of microstructure.

Meanwhile, Figure 2 (II) (A2, B2 and C2) displays the microstructure of filler metal VZ2250 at the Cu base and BAg-8 at the SS304 base. By referring to the figure, it is clear that there are voids at the base metal (Cu interface) and most notably at the porous area (after the diffusion zone) of A2 and C2. Besides, a fine micro-crack appears at the base metal (SS304 interface) of A2 and C2, but unnoticeable at B2. However, the joint microstructure is improved compared with the filler metal of BAg-8 for both sides. Besides, it was also discovered that with increasing the brazing temperature, the thickness of the diffusion zone appears to be increasing at both Cu and SS304 base and flowing towards porous Ni. Thus, microstructure particularly for C2 was preferred to further analyse the compositions of elements at both interfaces. It is remarkable to focus on the specimen brazed at 910 ˚C for 15 minutes of brazing time as for this specimen, the void areas are least.

Table 2: *Compositions of different points marked in Figure 3.*

Point	Elemental Compositions (at. %)			
	Cu	**Ni**	**Sn**	**P**
A	57.07	40.22	1.48	1.24
B	4.29	69.39	0.43	25.89
C	4.29	69.39	0.43	25.89
D	4.29	69.39	0.43	25.89

Table 3: *Compositions of different points marked in*

Point	Elemental Compositions (at. %)						
	Fe	**Cr**	**Ni**	**Si**	**Mn**	**C**	**Ag**
A	63.38	19.07	7.54	-	-	9.14	0.87
B	-	-	96.59	-	-	-	3.41
C	-	-	78.02	-	-	-	21.98
D	-	-	97.64	-	-	-	2.36

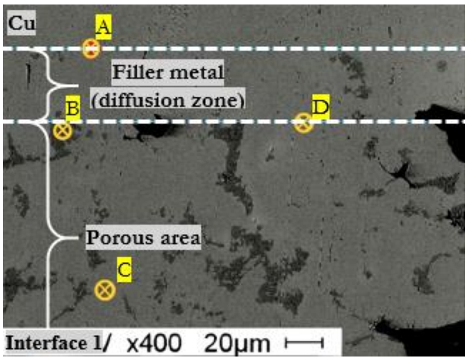

Figure 3: *Interface 1 of C2 (Cu/VZ2250/Porous Ni).*

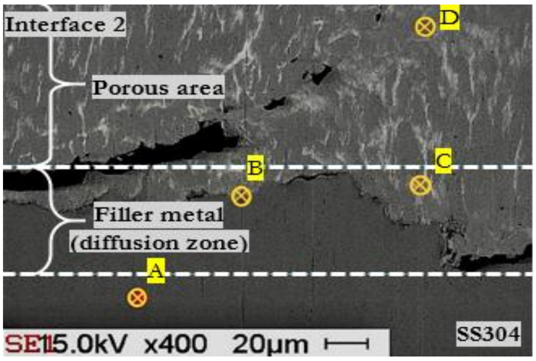

Figure 4: *Interface 2 of C2 (Porous Ni/BAg-8/SS304).*

FESEM equipped with EDS was used to examine the microstructure of the joint interface in order to identify possible existing filler metal elements. According to the EDS analysis, the major elements contributing to the formation of joining microstructure of Cu interface, are Ni and P elements. This is owing to the high weight percentage (wt. %) of Ni and P reported during the analysis of the different brazing temperatures. As can seen in Figure 3, Ni and P were found to diffuse at the diffusion zone towards the porous area. The EDS analysis (Table 2) confirmed that point B – D is rich in Ni and P elements and expected that Ni-P phase could emerge. Besides, point A is rich in Cu element which indicate the Cu base. Apart of that, it can be noticed that Ni element were found in the point A. This element appear from filler metal and porous Ni. Thus, it is expected that Cu-Ni phase could emerge.

Furthermore, the presence of element P in the filler metal tends to increase fluidity [19]. EDS analysis of different brazing temperatures showed that the wt. % of element P increasing as the brazing temperatures increased. Therefore, it can be concluded that this analyse is in agreement with the above discussion. P tends to accelerate the wetting of the filler metal and diffuse towards the porous Ni to create a strong bond.

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This statement can be agreed upon with shear strength analysis which will be discussed in the next sub-topic.

The EDS analysis of the SS304 base with filler metal BAg-8 are shown in Figure 4 and Table 3. The elemental compositions of SS304 are shown by point A. Due to the high concentration of Fe and Cr in this area, a Fe-Cr phase could emerge. Furthermore, it was discovered that Ni from filler metal diffused into SS304, as Ni has an average solubility with Fe. A Fe-Ni could semblance as the wt. % of this element is higher. Element Mn at the SS304 interface does not appear in the EDS analysis of C2 compare with the A2 and B2. Mn tends to increase the strength.

Point C is largely comprised of a bright coloured Ag-rich solid solution. It is remarkable that Ag is difficult to diffuse into SS304. The high amount of Ni at Point C might be the porous Ni area that is strongly influenced by the Ag element from the filler metal. Besides, it is also noticed that at brazing temperature of 910 ˚C, the diffusion of Ag was higher as compared with 830 ˚C and 870 ˚C. Additionally, due to the high diffusion of the filler metal during brazing, there is a small amount of Ag at point D, which is the area of Porous Ni. At all points of B, C and D, it can be seen that no diffusion occurred from the SS304 elements at 910 ˚C.

3.2 Shear Strength

The bonding strength before fracture was identified as the maximum shear strength value. Figure 5 shows the shear strength of the 2 interface, Cu/VZ2250/Porous Ni and Porous Ni/BAg-8/SS304 in relation to different brazing temperature. Furthermore, Dino-lite microscope captures and observes the fracture surface of the 2 interfaces.

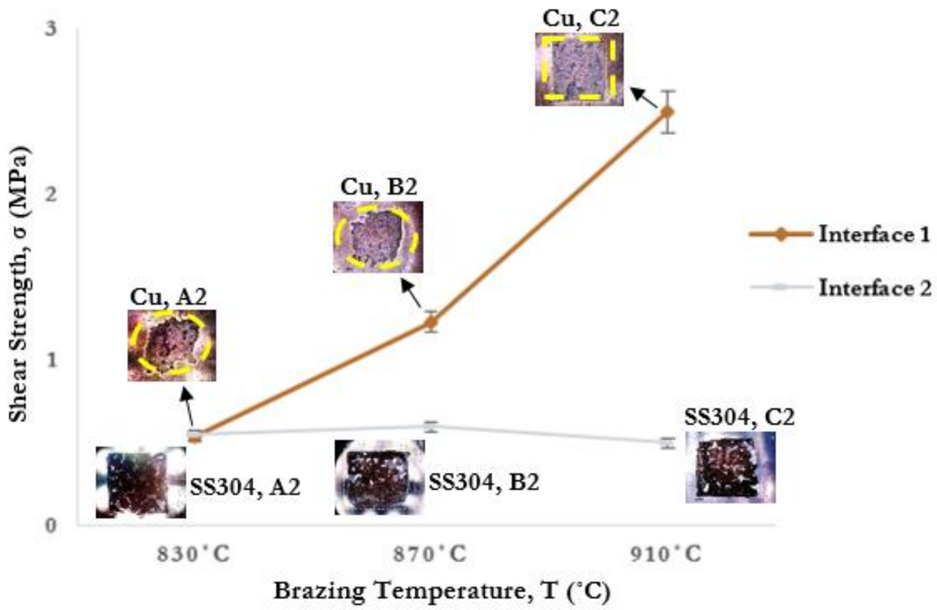

Figure 5: *The shear strength of brazed Cu/VZ2250/Porous Ni/BAg-8/SS304 at different temperature.*

The brazed joint strength of Cu/VZ2250/Porous Ni increased when the brazing temperature was increased above 830 ˚C. Therefore, the shear strength of Cu/VZ2250/Porous Ni was maximum at a brazing temperature of 910 ˚C with value of 2.50 MPa. The fracture surface is comparable for A2 and B2, as shown in Figure 5. However, the condition of the C2 fracture surface is slightly different. It can be noticed that the porous Ni that is attached to the Cu interface has more leftovers than the other two fracture surface (A2 & B2). The leftover of porous Ni that attached with Cu interface between porous Ni and Cu is relatively robust. However, the brazed joint strength of Porous Ni/BAg-8/SS304 decreased when the brazing temperature was increased above 830 ˚C. Hence, the shear strength of Porous Ni/BAg-8/SS304 was maximum at the brazing temperature of 870 ˚C with value of 0.60 MPa. Moreover, the micro-cracks pointedly (Figure 2 (I)) reduced shear strength and revealed a weak bonding. The fracture surface ruptured at the interface, as shown in C2. Thus, it is assumed that joint bonding of C2 is average compare with the A2 and B2.

From the data, it can be seen that the heat is adequate to ensure successful bonding at a brazing temperature of 830 ˚C until 910 ˚C. Yet, increasing the brazing temperature negotiated the strength of Porous Ni/BAg-8/SS304 due to the element Ag insoluble with Ni. This factor caused the deterioration of the shear strength as the brazing temperature was increased. It is assumed that the filler metal BAg-8 has diffused completely till the brazing temperature reaches 870 ˚C. As a conclusion, the obtained value of shear strength are comparable with the previous study [1].

One issue that frequently affects many types of heat exchangers is heat exchanger fouling. As a result, the heat transfer surface is affected and the rate of heat transfer through that surface is decreased overall. Fouling reduces the efficiency of heat exchanger and inhibits effective heat transfer. Therefore, shear strength that are applied to heat exchanger are less inclined to fouling. Due to the weeping effect, the high shear strength prevents deposits from forming on the plate surface. The relation of shear strength on deposit formation for crude oil fouling in a heat exchanger tube was investigated by Sampath and his team. They observed that the shear strength of the wall allows fouling precursors to be removed and reduces deposit accumulation [20]. Furthermore, the impact of surface shear strength on deposits removal was investigated further by Crittenden and his team. The fouling deposits can be mitigated from the surface by increasing wall shear strength [21]. Therefore, the current study's shear strength is assumed sufficient to withstand fouling prevention in a heat exchanger.

4 Conclusion

The current study showed the microstructures of brazed porous Ni to Cu and SS304 using different brazing filler metals. Overall, the results showed that brazed Cu/VZ2250/Porous Ni/BAg-8/SS304 acquired a good joint bonding compared with the brazed Cu/BAg-8/Porous Ni/BAg-8/SS304. Besides, brazed Cu/VZ2250/Porous Ni/BAg-8/SS304 tends to reduce formation of voids and cracks. Therefore, the brazed joints' strength was identified with the shear strength also the fracture surface of the 2 interfaces was observed and included in this study. Based on the data obtain, the brazed joint strength of Cu/VZ2250/Porous Ni increased when the brazing temperature increased above 830 ˚C and the maximum shear strength was obtained at a brazing temperature of 910 ˚C. The heat was sufficient to melt the filler and diffused towards porous Ni. Besides, element Ni and P was identified as the main element that contributing to the formation of joint bonding for Cu/VZ2250/Porous Ni. Within this identification, it is assumed that Ni-P phases would emerge. However, the brazed joint strength of Porous Ni/BAg-8/SS304 was decreased as the brazing temperature was increased. The maximum shear strength was obtained at a brazing temperature of 870 ˚C. The decreasing of shear strength for brazed Porous Ni/BAg-8/SS304 might causes by the element Ag in filler. As Ag insoluble with Ni, it tends to weaken the shear strength and deteriorate the bonding.

5 Declarations

5.1 Acknowledgements

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5.2 Competing Interests

There is no conflict of interest.

5.3 Publisher's Note

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