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Elements Diffusion of the Interface for Be/ 00Cr17Ni14Mo2 Stainless Steel with Cu Interlayer by Vacuum Diffusion Bonding

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Keywords: beryllium, 00Cr17Ni14Mo2 stainless steel, copper, diffusion bonding, element diffusion, interface.

Abstract. Diffusion bonding is an effective way to join beryllium with other materials. To study the elements diffusion on the bonding interface to improve the joints performance, the microstructure, and the distribution of composition and phase on the interface of Be/Cu/00Cr17Ni14Mo2 stainless steel diffusion bonding were analyzed using scanning electron microscopy (SEM), energy dispersive spectrometer (EDS), scanning auger microscopy (SAM), x-ray diffraction (XRD), and the elements diffusion on the interface and the relationship between phases distribution and microstructure were also discussed. The result show that the original interface of Be/Cu diffusion bonding is the most weak site of the whole diffusion bonding zone and the fracture site of specimen. Dissimilar metal atoms gather first in the grain boundary and form intermetallic compounds, and the grain boundary or intermetallic compounds are the main gateway of the elements diffusion. The copper interlayer has greatly hindered the interdiffusion between stainless steel elements and beryllium, so the formation of intermetallic compounds between beryllium and stainless steel elements are avoided effectively to improve the diffusion bonding joints strength.

Introduction

In these years, scholars carried on a lot of research on diffusion bonding of dissimilar metals, which focused mainly on diffusion bonding of dissimilar metals with[1-5] and without[6-8] an interlayer material. Beryllium is a reactor material, because it has the lowest Z, relatively high thermal conductivity, low activation, etc. Diffusion bonding is an effective way to join beryllium with other materials. With the process, joining of materials is achieved through atomic transport and mechanical processes at the bonding interface. It is difficult to bond beryllium with 00Cr17Ni14Mo2 stainless steel, because beryllium is very brittle, and beryllium with the main elements of 00Cr17Ni14Mo2 stainless steel easily form brittle intermetallic compounds in the process of diffusion bonding. Therefore, copper interlayer between beryllium and 00Cr17Ni14Mo2 stainless steel is used to reduce the mutual diffusion between beryllium and stainless steel eliments, and the formation of brittle intermetallic compounds. In this paper, the elements diffusion on the interface of Be/Cu/ 00Cr17Ni14Mo2 stainless steel diffusion bonding in terms of the microstructure and the distribution of elements and phases have been investigated, and good joint can be obtained using copper as an interlayer material.

Experimental Procedure

Materials and Specimens

Beryllium specimens are made of hot-pressed beryllium, which composition show in Table 1. The 00Cr17Ni14Mo2 stainless steel (SS) is the austenite anti- hydrogen stainless steel and the impurity total contents is less than 0.17%, which the main alloying elements content show in Table 2. Bars of Beryllium and SS are 10 mm in diameter and 5 mm in height. Diffusion bonding was achieved by

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hot pressing at 50MPa and 750°C in 10^{-3} Pa vacuum, and the holding time is 2h (750°C/50MPa/2h). copper foil of 50 μm in thickness is used as interlayers。

Table1. The content of heat pressed Be powder, w/%

Be	Fe	Al	Ni	Cr	Mn	Pb	Mg	O
Bal.	0.18	0.078	0.003	0.01	0.013	0.001	0.12	0.75

Table2. The content of SS, w/%

Fe	Cr	Ni	Mo	Mn
Bal.	17.2	14.27	2.19	1.05

Instruments and Experiment Method

The microstructure morphologies were recorded using TESCAN VEGA3 SBH scanning electron microscopy (SEM). The composition of the diffusion bonding zone were recorded using BRUKER QUANTAX 100 energy dispersive spectrometer (EDS). The XRD pattern were recorded using D/MAX-1200 X-ray diffraction, at a Cu-anode, 40kV tube voltage, 30mA tube current and 2° min^{-1} scanning speed. The SAM spectra were recorded using PHI650SAM scanning auger microscopy, at 3kV electron excitation energy, 0.25% energy resolution, 100 nA electron beam current. A tensile tester was used for testing the mechanical properties of the joints.

Results and Discussion

Experimental

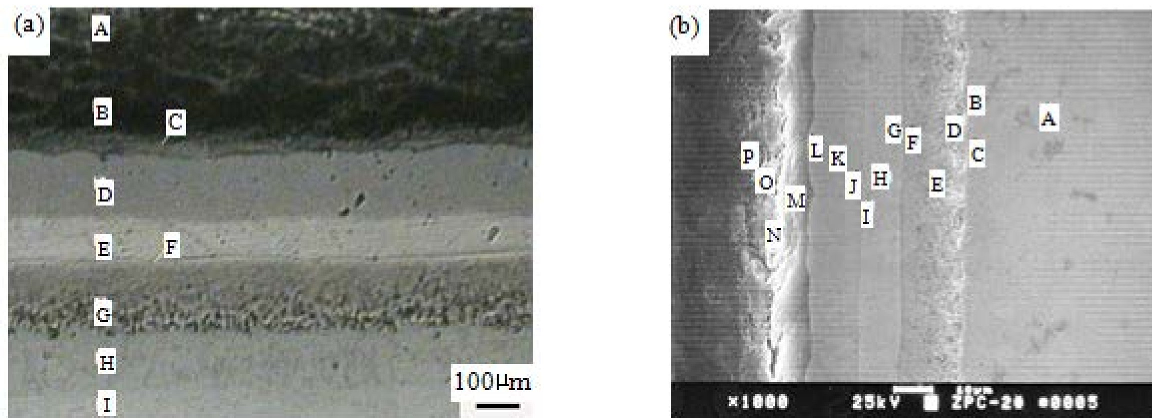

Fig.1 The microstructure of joint zone. (a) Metallurgical micrograph, (b) SEM micrograph.

The microstructure of Be/Cu/00Cr17Ni14Mo2 stainless steel diffusion bonding joint show in Fig. 1. Fig. 1 (a) shows that the diffusion bonding zone includes A, B, C, D, E, F, G, H and I layer, which are almost parallel and perpendicular to the outside forces direction. The results of EDS testing each zone in Fig.1 (b) show in Table 3, which the contents of chromium, copper, molybdenum and nickel are about 19%, 10%, 4% and 2% in the DEF layer (SS side), respectively, and some phases are corroded to pit by aqueous nitric acid. There are about 87% copper and 9% nickel in the GHI layer, which shows good corrosion resistance. Becauase copper and nickel are face centered cubic lattice and the lattice constants are similar, they can form the infinite displacement solid solution for corrosion resistance. The copper content is increased (average 93%) and the nickel content is reduced in the JKL layer, which also shows excellent corrosion resistance. Because the energy dispersive spectrometer (EDS) can not detect beryllium, the main element is copper in the MNOP layer (beryllium side), and it is reason that the elements content are the relative in the beryllium side.

Therefore, Fig.1 b shows that A part is the SS matrix, EF layer and BCD layer are respectively diffusion zone and diffusion transition zone in the SS side, GHIJKL layer is the intermediate bonding region (the original copper interlayer), MNO layer is the diffusion bonding zone between the original copper interlayer and beryllium, P part is diffusion zone in the beryllium side, The D/C interface is the boundary between the diffusion transition zone and the SS matrix in the SS side, The G/F interface is the diffusion bonding boundary between the original copper interlayer and the diffusion zone in the SS side, and The J/I interface is the boundary of different diffusion belt in the original copper interlayer.

The fracture morphologies of the diffusion bonding joint for Be/Cu/00Cr17Ni14Mo2 stainless steel show in Fig. 2, which intermetallic compounds grow up along the grain boundary and form finally a three-dimensional network structure, and there are microcracks or grooves in the grain boundaries, it is reason that the specimen are the brittle fracture on the grain boundary. The XRD patterns of stripping layer from the SS side fracture to the SS matrix and from the beryllium side fracture to the beryllium matrix are shown in Fig. 3 and in Fig. 4, respectively. The results of XRD phase analysis show that the fracture is brittle α-Be (beryllium base solid solution) as matrix in the beryllium side, and the brittle Be_2Fe and Be_2Cu are distributed along the grain boundaries, while the fracture is brittle BeCu as matrix in the SS side, and the brittle Be_2Fe and Be_2Cu are distributed along the grain boundaries.

Table3.The EDS testing results of Fe, Cr, Ni, Mo and Cu in diffusing bonding zone，w/ %

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P
Fe	66.7	58	72	69	66	62	3.5	2.8	2.5	3.7	3.1	2.6	1.6	1.4		
Cr	18.2	14	18	20	18	19	0.6	0.5	0.2	0.3	0.2	0.1	0.2	0.1		
Ni	13.2	25	7.6	2.3	2.1	2.1	9.5	9.3	8.6	4.6	2.7	0.7	0.7	0.8	0.6	
Mo	1.66	0.4	0.4	4.8	3.4	4.6	0.0	0.1	0.0	0.0	0.0	0.0	1.2	1.01	0.5	0.3
Cu	0.00	1.7	1.4	4.8	10	12	86	87	89	91	94	97	96	97	98	99

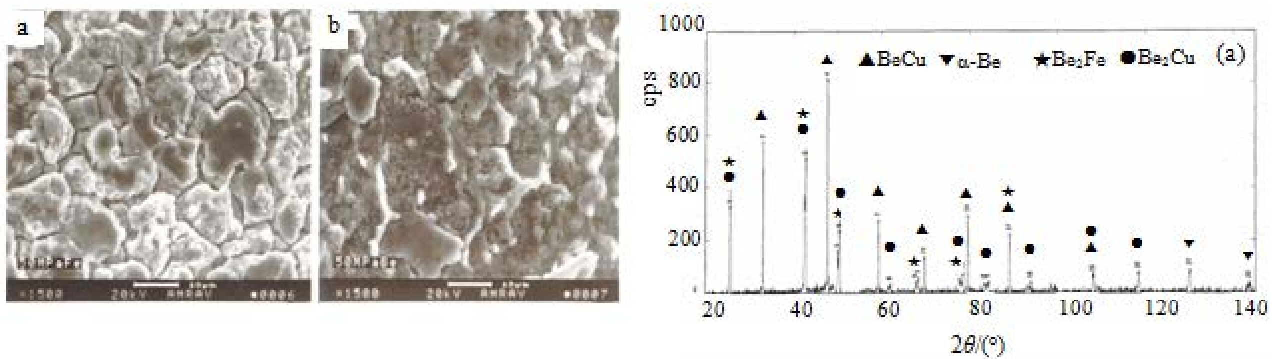

Fig.2 The fracture morphology of joint. (a) SS side, (b) Be side

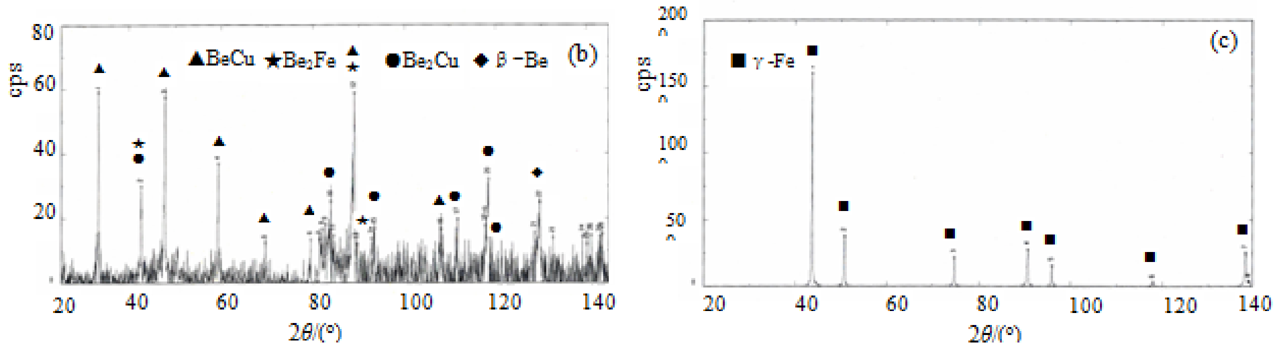

Fig. 3 The XRD patterns of the SS side fracture and after peeling layer.(a) SS side fracture, (b) after peeling 45μm, (c) after peeling 60μm

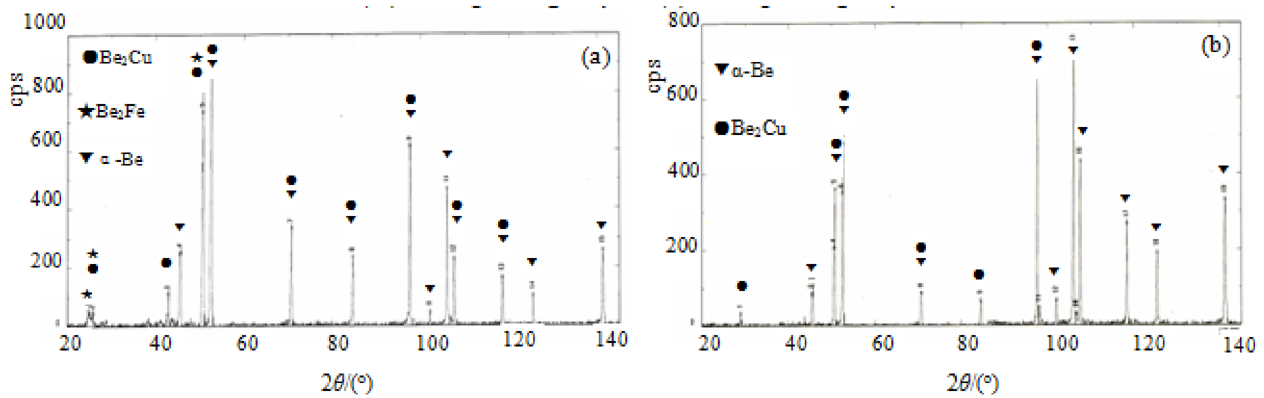

Fig. 4 The XRD patterns of the Be side fracture and after peeling layer.(a) Be side, (b) after peeling 40μm

In Be/Cu/ 00Cr17Ni14Mo2 diffusion bonding process, first of all are the interdiffusion between Be←→Cu and Fe←→Cu, then lead to the interdiffusion between Be and Fe. The Cu-Fe binary phase diagram[9] shows that Cu and Fe can not form intermetallic compounds, and can only form small amount of solid solution with very low solid solubility under the condition of temperature 800℃. Due to the concentration gradients of Fe and Cu are very large on the Cu/SS interface, which promote the transfer of Cu to the SS and Fe to the copper interlayer, and Fe further spread to the beryllium side and result in forming Be_2Fe. Fig. 3 show that the diffraction intensities of Be_2Fe, Be_2Cu and BeCu are decreased with the increase of the distance from the SS side fracture, which the lowest is at 45μm peeled from the SS side fracture and the background of spectral line is enhanced. Based on XRD principle, the phases diffraction intensity will decrease and the background intensity will increase when crystal lattice is distorted. Therefore, with temperature decrease after hot pressing, Fe and Cu are precipitated from the over saturated solid solution and lead to serious lattice distortion[10]. Fig. 3 c shows that the main phase is γ-Fe at 60μm peeled from the SS side fracture, which explains that serious lattice distortion area is at the bonding zone of copper and SS. The Be-Cu binary phase diagram[9] shows that the solid solubility of copper in α-Be matrix and the solid solubility of beryllium in copper matrix are about 6% and 12% under the condition of temperature 800℃, respectively, and Be_2Cu and BeCu are formed in turn with copper content increasing. Therefore, we can infer that Be_2Cu and BeCu formed in the copper side of the Be/Cu interface, and BeCu is the matrix. The beryllium based solid solution with copper is formed first, then its over saturated copper with beryllium form Be_2Cu in the beryllium side of the Be/Cu interface, and it is difficult to form BeCu. These inferences are consistent with the XRD analysis results in Fig. 3 and Fig. 4. Because the beryllium solid solubility is about 12% in copper matrix, the mutual diffusion ability between copper and beryllium is good and lead to form Be_2Fe in the SS side. On the other hand, the formation of Be_2Cu and BeCu improve diffusion ability of copper to beryllium matrix and beryllium to copper interlayer[11], and it is more easily to beryllium diffuse

into the SS side and form Be_2Fe in the SS side. With the decrease of temperature after hot pressing, the over saturation beryllium and copper are precipitated from the copper based solid solution and the beryllium based solid solution, respectively, and lead to lattice distortion in the original copper interlay region and the beryllium side. Due to solubility of copper in beryllium matrix is lower than one of beryllium in copper matrix, the lattice distortion degree in the beryllium side is more serious than one in the original copper interlay region. Fig.5 show the distributions of Fe, Be, Cu, Cr and Ni near the Be/Cu/SS bonding interface measured using AES, which copper interlayer greatly hindered the Fe, Cr and Ni into the beryllium side, thus prevent $Be_{21}Ni_5$, $Be_{12}Cr$ and NiBe etc.forming in beryllium matrix, so the formation of intermetallic compounds between beryllium and stainless steel element are avoided effectively to improve the diffusion welding performance.

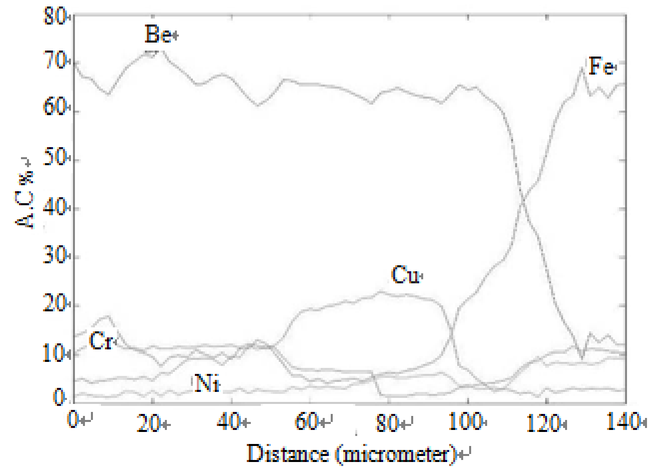

Fig.5 The element distribution at the interface of Bc/Cu/SS diffusion bonding

Therefore, both the original copper interlayer and the interface region of SS side are BeCu as matrix and Be_2Cu, Be_2Fe distribute on BeCu grain boundary, so their structures are basically the same. It is reason that the bonding ability of the interface between the original copper interlayer and the SS side (Cu/SS interface) is good and the interface is combined closely. The interface region of beryllium side is α-Be (beryllium base solid solution) as matrix and Be_2Cu, Be_2Fe distribute on α-Be grain boundary, which make a great difference with the structure of the original copper interlayer. The lattice static distortion and the forming phases between the original copper interlayer and the interface region of the beryllium side are very different, which causing different volume change produces large internal stress field. It is reason that the interface between the original copper interlayer and the beryllium side (Cu/Be interface) is the most weak site of the whole diffusion bonding zone and the fracture site of specimen.

The test results of tensile strength show that the tensile strength of Be/00Cr17Ni14Mo2 stainless steel was 12MPa, while the tensile strength of Be/Cu/00Cr17Ni14Mo2 stainless steel was 60 MPa.

Conclusion

1) During Be/Cu/00Cr17Ni14Mo2 stainless steel diffusion bonding, dissimilar metal atoms first gather and form intermetallic compounds on the grain boundaries, which causing stress concentration and the microcrack are the main reason why reduce the diffusion bonding performance.
2) During Be/Cu/00Cr17Ni14Mo2 stainless steel diffusion bonding, the crystal structure exist in a great difference on the both sides of the Be/Cu interface, and there is large internal stress on the Be/Cu interface zone, so the Be/Cu interface is the most weak site of the whole diffusion bonding zone and fracture site of specimen.

3) The copper interlayer has greatly hindered the diffusion of Fe and other alloy elements to the beryllium side, and reduced the formation of intermetallic compounds and improved the joint performance.

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