Destructive and non-destructive evaluation of cu/cu diffusion bonding with interlayer aluminum

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Abstract
The current study is established an inspection procedure for assessing quality of diffusion bonded joints using destructive and non-destructive method. Diffusion bonding of commercially pure copper with aluminium interlayer was carried out uniaxial load at 15MPa for different temperatures under holding time 60 min in vacuum atmosphere. The bond qualities were determined by destructive and non-destructive testing method (ultrasonic C-scan). The bond interface and bonded samples were analysed using optical and scanning electron microscopy (SEM). The element composition of the fractured and bonded area is determined using the Energy Dispersive Spectrometry (EDS). The bond quality obtained by both testing methods and its parameters are correlated. The optimized bonding parameter for best bonding characteristics for copper diffusion bonding with aluminum interlayer is reported.

1. Introduction:
Diffusion bonding is a bonding process where the primary mechanism is interdiffusion of atoms throughout the interlayer interface to the parent metal [1]. It’s the only success full cross sectional bonding, it is also of in internal modification, can be obtained. Basically, there is no liquid state and the monolithic compound is form complete under solid state conditions. A similar process in liquid phase is transient liquid phase (TLP) diffusion bonding in which a suitable interlayer is placed between the parent metal to be joined and interdiffusion between the interlayer and the base material leads to the formation of a low melting point liquid phase at the bonding temperature. The interlayer liquid must be a low melting alloy will diffuse rapidly into the base metal. As this element diffuses away, the interfacial region becomes enriched in the non-diffusing elements; it will solidify due to loss into the bulk of the diffusing component. In other words, TLP bonding starts and ends as a diffusion bond and it reduces pressure and time required for bonding. In the optimized condition, mechanical and metallurgical properties across the joined layer are comparable to the parent metal, Due to heating and isothermal cooling of the whole sample.

Diffusion bonding is done to surface prepared and after joining at elevated temperature ranging from 0.5 to 0.8 Tm (where Tm is the absolute melting point of parent metal) under certain pressure which is mostly fraction of yield strength and so there is no macroscopic deformation. Metallographic studies on the bonding will be estimate the area fraction bonded (Afb) with unbonded area and bonded area distribution in metallographic studies. The quality and durability are be evaluated by non-destructive test will be able to give interface interlayer properties of diffusion bonded joints without any destruction. The non-destructive testing is done by ultrasonic method, because it’s more suitable to find interface bonding.
As well as conventionally the bond reliability of bonds to be evaluated by the destructive tests such as tensile strength, micro hardness throughout the interlayer interface, The strength ratio is determined from the tensile test (ratio between the room temperature tensile strength of bonds to the room temperature of the tensile strength of a unbonded material subjected to the same thermal conditions as those of the bonded samples) [2]. Ultrasonic C-scan testing method is most suitable to evaluate the diffusion bond interlayer interface. It is possible to visualize that flaw size and their distribution by using this method. Comparing both non-destructive testing results (reflection coefficient) with destructive testing results (strength ratio).

2. Experimental work:
In the study diffusion bonding, copper and aluminium as a parent material and interlayer respectively. Round extruded commercially pure bars of two metals as specimens (40mm diameter x 20mm length). The specimens are surface finish by joining side of copper specimen and aluminium interlayer as both sides was polished by using rotary silicon carbide papers 400, 600, 800, 1000, 1200 grit emery papers respectively. After mirror finished by cloth polishing and diamond polishing for fine finishing and cleaned with acetone before performing the experiment. However, this surface preparation does not prevent the formation of Al$_2$O$_3$ layer but we can reduce the thickness of the layer, which plays an important role in joint formation.

![Figure 1. Diffusion bonding furnace with hydraulic load equipment](image)

Diffusion bonding was performed in the diffusion bonding setup which is shown in the picture in Figure 1. Constructed by alloy steel gives creep properties, which is not deformed at the high bonding temperature. The initial load applied by top cap and the main load applied by the hydraulic press with is having a wide range of up to 100 tons with accuracy ±100 N. Vacuum pump is connected to the furnace which rotary pump, was used to evacuate the chamber down to 10$^{-4}$mbar. Setup of the furnace is well connected with cooling unit with temperature durable pipes to cool the furnace by water for isothermal cooling stage with cooling rate 2°C/min. Specimens are fixed in dies in heating chamber. Experiments are conducted with various process parameters, mainly temperature in rage of 450-625°C.

3. Destructive testing (tensile testing and micro hardness):
The bonded samples are testing by using non-destructive testing ultrasonic C scan method for various process parameter conditions. The bonded samples were transversely cut by EDM through the bond
interface for destructive testing such as micro hardness and micro tensile testing. For bonding interface and voids at some parameters were observed in an unetched condition of interlayer bonded samples by scanning electron microscopic testing. Bonded samples are examined chemical compositions at the interlayer interfaces to find various phases formed by bonding.

Tensile, hardness such as destructive testing is done for strength & quality of bond examinations. Cylindrical specimen as dimensions 6 mm in diameter and 40 mm length for tensile testing performed by Tinius Olsen H10k S-series testing machine at room temperature. Strength ratio is considered as parameter of quality of bond was evaluated by the tensile strength results of bond. Micro Vickers hardness survey was made on interlayer bond interface done in various regions (base material, interface layer and aluminium layer). The survey was conducted by using micro Vickers hardness tester with load 200gm with dwell time of 10 sec.

4. Non-destructive testing (ultrasonic C-scan method) evaluation:
Diffusion bonding quality is examined by non-destructive testing ultrasonic testing method with the scan charts of A, B and C scan. A-scan chart shows echo signal coming from corresponding scanning place and respective ‘C’ scan images of perfectly bonded and imperfectly bonded samples. Perfect bonding resembles maximum back wall echo, small reflection from interlayer region because of shear velocity change in aluminium and copper. For imperfectly bonding back wall echo will be less, in interface region echo will come back due to imperfect bond [2,3].

Figure. 2 shows output of C-scan for the bonded samples at respective process parameters bonding done. In C-scan the scan shown difference of bond in colour coding, the bond quality was examined and analysed by colour variation in ultrasonic C-scan results by analyser software by that we will calculate reflection coefficient. Blue colour indicates that the very less or none intensity of the reflected wave from the bond interlayer interface (Figure 2a), completely blue means full cross-sectional bonding is done. Red colour indicates that maximum intensity in the scan (Figure 2c), its reflected echo from interlayer area and it’s clear no bonding is done there. Intermediate colours are shown partial bonding between metal and interlayer.

**Figure. 2 a) C-scan image at 625°C, 15MPa and 60min, b) C-scan image of 600°C, 15MPa and 60min c) C-scan image of unbonded sample**

Bond quality is normally determined by strength ratio. The strength ratio is determined from the tensile test (ratio between the room temperature tensile strength of bonds to room temperature of tensile strength of a unbonded material subjected to the same thermal conditions as those of the bonded samples) [2,3] Table 1 also indicates the determined values of strength ratio.

Figure. 3a shows the relation between strength ratio and the reflection coefficient. The reflection coefficient increases as the strength ratio decreases indicate the quality of bond it correlates non-destructive parameters (reflection coefficient) and the strength ratio resulted due to destructive
testing. Figure 3b is shown reflection coefficient increases as area fraction bonded decreases or bond quality is decreasing [7].

Table 1. Effect of diffusion bonding process parameters on quality

| Sample no. | Bonding temperature \(^{\circ}\text{C}\) | Bonding pressure (MPa) | Holding time (min) | Area of bonding fraction \(A_r\) | Strength ratio | Reflection coefficient \(R_f\) |
|------------|--------------------------------|------------------------|-------------------|-----------------|----------------|-----------------------------|
| 1          | 500                           | 15                     | 60                | 0.40            | 0.20           | 0.8214                     |
| 2          | 550                           | 15                     | 60                | 0.57            | 0.32           | 0.6410                     |
| 3          | 575                           | 15                     | 60                | 0.68            | 0.40           | 0.5197                     |
| 4          | 600                           | 15                     | 60                | 0.79            | 0.47           | 0.4223                     |
| 5          | 625                           | 15                     | 60                | 0.98            | 0.61           | 0.2181                     |

Figure 3. a) Change of strength ratio to reflection coefficient, b) Change of reflection coefficient as a function of area fraction bond

Scanning electronic microscopic (SEM) and Electronic dispersive spectroscopy (EDS) analysis were done to analysis the phase formation on the interface between copper and aluminium interlayer bonded samples. The SEM micrographs, shown in Figure 4., discloses distinct phases of the bonded interface at 625\(^{\circ}\)C as 60 min holding time under 15 MPa. Thickness of the interface zone is measured to be 20 micrometers, is the lowest thickness of all process parameters done. Concentration of chemical components along the interface is at high temperature ascribe to the formation of assorted the brittle intermetallic compounds [8]. It is analysed by EDS throughout interface joint. It conforms AlCu\(_3\), Al\(_3\)Cu\(_9\) the intermetallic formation at the interface. The micro hardness measured in the intermetallic regions in order

Another destructive test, micro Vickers hardness is examined for 600\(^\circ\)C and 650\(^\circ\)C samples etched with 100ml distilled water and 5ml of HNO\(_3\) solution, for interlayer examination and conducting hardness test on various concentration regions of intermetallics copper side average hardness 72 HV0.2, at copper – aluminium joint layer average hardness 130HV0.2 because copper rich intermetallics formation like AlCu\(_4\), Al\(_3\)Cu\(_9\), and in the first layer of the intermetallic layer has highest than other layer about 282HV0.2 because of hard intermetallics like AlCu\(_3\), Al\(_3\)Cu\(_9\).
5. Conclusion:

The diffusion bonding of similar material with interlayer was investigated. Subsequent results were obtained: The diffusion bonding of Cu/Cu with interlayer Al is not done below 450°C at any pressure, at 625°C, 60min and 15MPa best results were recorded. Nondestructive ultrasonic test used to evaluation of bonding. Correlation methodology between nondestructive parameters to destructive parameters was evaluated. With the help of SEM and EDS elemental composition and intermetallics reported throughout the interface layers.

Reference:

[1] MacDonald W.D. and Eagar T.W. (1992), The Metal Science of Joining 93-100, ed. Cieslak M.J. et al., The Minerals, Metals & Materials Society.
[2] Suresh Kumar. S, B. Ravisankar. (2014), Applied Mechanics and Materials Vols. 592-594 (2014) pp 289-293
[3] S. Sureshkumar, J. Krishnamoorthy, B. Ravisankar, P.C. Angelo, Indian journal of Engineering and materials sciences, Vol 16, pp 331-334,(2009).
[4] EvrenAtasoy, NizamettinKahraman, Materials Characterization 59 (2008) 1481–1490.
[5] MitsuakiKoath, KazumasaNishio, Tomiko Yamaguchi, NDT& E International 35 (2002) 263-271.
[6] K. Milne, P. Cawley, P.B. Nagy, D.C. Wright, A. Dunhil, *J Nondestruct Eval* (2011) 30:225–236
[7] G. Mahendran & V. Balasubramanian & T. Senthilvela, *Int J Adv Manuf Technol* (2009) 42:689–695
[8] F. A. Calvo, A. Urena, J. M. Gomez De Salazar, F. Mollleda, *Journal of materials science* 23 (1988) 2273-2280