Journal of Manufacturing Processes 23 (2016) 13–20

Contents lists available at ScienceDirect

Journal of Manufacturing Processes

journal homepage: www.elsevier.com/locate/manpro

Technical Paper

Destructive and non-destructive evaluation of copper diffusion bonds

S. Suresh Kumar*, B. Ravisankar

Department of Metallurgical and Materials Engineering, National Institute of Technology, Tiruchirappalli 620 015, India

ARTICLE INFO

Article history:
Received 4 November 2015
Received in revised form 19 May 2016
Accepted 22 May 2016
Available online 7 June 2016

Keywords:
Diffusion bond
Copper
Interface quality
Grain growth
Pilling's model
Ultrasonic 'C' scan testing method

ABSTRACT

The current study reports diffusion bonding phenomena of copper to copper (Cu/Cu). The bonding was conducted in the temperature range varying between 700 °C and 850 °C at varying soaking time (30–120 min) under uniaxial pressure of 5–15 MPa in vacuum atmosphere. The bonded regions were characterised, using light and scanning electron microscopic (SEM) studies. The bonded strength was evaluated through destructive testing using micro tensile and microhardness tests. In addition, the bond quality was also assessed by nondestructive testing (ultrasonic C-scan test) method. The grain growth equation was used to understand the bonding mechanism. The investigational results were compared with the model developed by Pilling, observing void closure by creep flow and diffusion mass transfer. The superior bond strength ratio of 91% was observed at bonding temperature of 750 °C for the applied pressure of 15 MPa of 60 min soaking period. The correlation between destructive and non-destructive tests was studied.

1. Introduction

Diffusion bonding is one of the solid-state welding processes. As its name implies, it involves the inter diffusion of atoms of similar or dissimilar materials to be joined in the solid state. Sometimes it can also be done at a liquid state (when a molten interlayer is used) [1]. The process parameters such as applied pressure, temperature, soaking time should be optimised for the rapid atomic movements in the bonding interphase, which result in a pore less bonding. The widely used resistance and fusion welding processes cannot overcome the in compatibility barrier (oxide layer) inherent in metals and alloys. These barriers are avoided using vacuum diffusion bonding [2]. Diffusion bonding can join similar, dissimilar, composites and ceramic materials. Diffusion bonds are heat-resistant, vacuum-tight, and vibration-proof with its products, retaining their high accuracy, dimensions and shape. The process does not involve macroscopic deformation. Diffusion bonding in vacuum does not require any expensive fluxes, electrodes and shielding gases. A weldment needs no subsequent machining as the process leaves no slag, flash or scale, nor is there any associated loss of metals. Diffusion bonding has contributed much to the advances in the development of aircraft, missile, electronics, nuclear, aerospace, chemicals and gas fields [3].

Conventionally, the quality of the diffusion bonds is estimated by microscopic characterisation and destructive tests such as hardness measurement across the bond interface and tensile strength etc., then to be estimated with that of base metal properties [4]. Micrographs study on diffusion bonded joints is considered to be a capable tool to calculate the pore size and distribution in two dimensions as well as the area fraction bonded (AF) [5]. Non-destructive testing method (NDT) is used to detect and analyse the quality of bonded sample and interface strength of the diffusion bonded samples. Among all NDT methods, the ultrasonic C-scan testing method is the most efficient and suitable one for diffusion bonded joints evaluation [6]. Ultrasonic testing method can detect the unbonded islands effectively. But it is not suitable to quantify the degree of bond weakness.

The C-scan presentation provides a planar (top view) type view of the location and size of the test sample features [7]. Although the bonded and debonded areas could be differentiated by the method, there was a small quantitative survey of the C scan images to show the bonding grade. Furthermore, it will increase the sensitivity of flaws and detect the micro defects inside the materials. The high frequency method is more accommodating [8]. It is possible to calculate reflection coefficient and area fraction bonded (AF) with the help of ultrasonic A, B and C-scan method and also to envision that defective dimension and their distribution, by using this method [9,10].

In this paper, Cu–Cu bond interface was investigated using ultrasonic C-scan imaging technique, operating at a probe frequency of 5 MHz. The image processing results based on the completely

* Corresponding author. Tel.: +91 9345100464; fax: +91 431 2500 133.
E-mail addresses: ssureshrec@gmail.com (S. Suresh Kumar), brs@nitt.edu (B. Ravisankar).

http://dx.doi.org/10.1016/j.jmapro.2016.05.012
1526-6125/

Nomenclature

AF	area fraction bonded
AR	acoustic constraint of the reflected echo
AU	acoustic constraint of the incident echo
CP	commercial pure
D	grain diameter
dfh/dt	void closure rate
fh	fractional area of non-bonded interface
Q	activation energy
R	universal gas constant
R_C	reflection coefficient
n	stress exponent
σ	bonding pressure
T_M	absolute melting point
GBD	grain boundary diffusion
K_0	pre-exponential constant

bonded and non-bonded regions were found; the area fraction and reflection coefficient of the bond was estimated and verified from 'A' scan results [11]. From this study, the diffusion bonding parameters are optimised using the destructive test and non-destructive test. Very few research works were performed on the soundness of the bond joint and other features with the help of ultrasonic C-scan [12].

The main goal of this study is to establish details about the bond interface (various bonded/debonded areas) and its association with the quality of bonding, determined from destructive and non-destructive testing methods [13–15]. The diffusion bonding between the Cu/Cu similar systems is performed and the activation energy is calculated from the Arrhenius relationship. The experimental observations of the Cu/Cu system are also compared with the Pilling's model (Pilling, 1988) to understand the void closure property during the diffusion event [16,20].

2. Experimental procedure

In the current investigation, the commercial grade extruded copper is used for diffusion bonding experimentation. The chemical composition of commercial Cu material is shown in Table 1. The CP copper material properties used to estimate the theoretical void closure rates are tabulated in Table 2. The samples were prepared with 50 mm diameter and 20 mm length of surface finish 10 μm. Prior to the experimentation, the specimens were cleaned and degreased using acetone in an ultrasonic bath. The in-house designed diffusion bonding chamber equipped with a vacuum resistance heating unit is employed to conduct the experiments. It is shown in Fig. 1. Copper with copper was heated from room temperature to the predetermined bonding temperature at the heating rate of 25 °C min^{-1}. The experimentations were conducted with varying process parameters such as temperature, pressure and time. The process parameters were varied at the constant vacuum atmosphere (pressure of 10^{-3} mm Hg). It is summarised in Table 3. The samples were cooled in the furnace till it attained the room temperature.

Table 1
Chemical composition (in wt.%) of commercially pure copper.

Elements	Al	Fe	Cu	B	Zn	O
Composition (wt.%)	0.14	0.007	Bal.	0.018	0.09	0.092

Table 2
Material properties of commercially pure copper [19].

Properties	Values
Atomic volume (m^3)	1.18×10^{-29}
Pre-exponential for grain boundary diffusion (m^3 s^{-1})	5.0×10^{-15}
Activation energy for grain boundary diffusion (kJ mol^{-1})	104
Pre-exponential for core diffusion (m^3 s^{-1})	1.0×10^{-24}
Activation energy for core diffusion (kJ mol^{-1})	117
Stress exponent	4.8

Initially, the bonded samples quality was tested using ultrasonically test using C-scan method, processed at varying process conditions. Then the bonded samples were perpendicularly sliced, using EDM process (Fig. 2) for other destructive tests such as micro tensile and micro hardness. The cross sectional bonded Cu–Cu regions was polished to 3 μm surface finish and it was electrolytically etched in a solution of 50 ml pure water and 50 ml HNO_3 for micrographs evolution. The structural evolutions such as grain size and grain boundary of the bonded layer were found from the scanning electron microscopic (Hitachi, Japan, Model no. S3000H) studies. The bonding interface and the voids were noticed in an unetched condition in the bonded samples. The average grain size of the bonded samples was measured from the linear intercept method.

2.1. Destructive tests (tensile and hardness)

Tensile testing of the similar Cu/Cu bonding interface was performed by Tinius Olsen H10k S-series testing machine at room temperature with crosshead speed of 2 mm min^{-1}. The cylindrical specimen dimension is of 5 mm in diameter and 40 mm in length. The tests were conducted on both bonded and unbonded specimens processed at the same temperature conditions to evaluate the strength ratio. The strength ratio reflects the characteristics of the interphase and the quality of bond. The hardness survey was made on the bonded samples at different regions (base material and bonded region) to understand the effect of process parameters (temperature, pressure and soaking time) on structural evolution on its strength. The tests were conducted using Vickers hardness tester (Matsuzawa micro hardness tester, Japan, Model no. MMTX7) with the load of 15 g with dwell time of 20 s.

2.2. Mechanism of bonding

2.2.1. Grain growth kinetics

Diffusion behaviour of commercially pure Cu/Cu atom migration and grain growth are observed for different temperature conditions. Therefore, diffusion behaviour of Cu on Cu, diffusion and activation energy were computed and discussed as below.

The activation energy of the controlling mechanism was also calculated from the grain growth kinetics relation given by the following relation [16,21],

$$D_t^n - D_o^n = K_0 \exp\left(\frac{-Q}{RT}\right) \quad (1)$$

where $n = 2$, D_o and D_t are the as-pressed grain size ($t = 0$) and the grain size after the sample is bonded for time 't' respectively, K_0 is the pre-exponential constant, R is the universal gas constant, T is the temperature and Q is the effective activation energy. Eq. (1) can be written as follows,

$$\ln D = \ln K_0 + \left(\frac{-Q}{RT}\right) \quad (2)$$