STRENGTH AND ULTRASONIC CHARACTERIZATION OF METALLIC INTERFACES

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INTRODUCTION

In recent years, the process of diffusion bonding has found considerable usage in both the nuclear power and aerospace industries. This process requires the compression of mating surfaces at an elevated temperature for a given time. If optimum conditions of time, temperature, pressure and surface cleanliness are achieved, diffusion of material across the interface will occur, yielding interfacial mechanical properties identical to those of the bulk material. The use of insufficient bonding conditions may result in void formation, precipitation of undesired phases or lack of grain growth across the interface. The consequence will be an interface that is less than fully bonded, which will result in severe degradation of the mechanical properties. Applications of diffusion bonding to nuclear reactor fuel elements, helicopter rotor hubs, jet engine turbine blades, etc., thus make the ability to characterize the strength of these interfaces highly desirable.

Recent experimental results have shown that the ultrasonic reflectivity and transmissivity of an interface can be used to investigate details of the interface. In one application [1], such measurements have been used to determine the radial stress component in a shrink-fit coupler. In other applications, related techniques have been proven useful in characterizing the state of closure of a fatigue crack [2] and the weld quality in a pinch welded tube [3]. Regalbuto [4] applied pulse-echo measurements to the study of diffusion bonded joints with initial success. Theoretical models have also been developed that explain empirical results on partially contacting interfaces [5,6,7]. These models attribute the acoustic reflectivity of the interface to asperities in contact between the two surfaces.

In the following, the application of ultrasonic reflectivity for characterization of the quality of a variety of copper diffusion bonds is described; this is a continuation of previous work [8]. The quality of each diffusion bonded specimen also has been characterized by its ultimate engineering stress. Empirical correlations between reflection coefficient and ultimate stress have thus been obtained. Furthermore, fractography of the (failed) bonds provided information on the relative fraction of bonded areas. The latter information is useful in testing a "distributed spring" model [6], which makes specific statements on the relation between contacts and the expected reflection coefficient. It was found that the theory describes the experimental observations quantitatively.

EXPERIMENTAL PROCEDURE

Sample Preparation

Disks of 99.99% copper, one inch in diameter and one-half inch in thickness were used to produce the diffusion bonds. These disks were mechanically polished flat and given a final chemical polish (55% nitric acid, 25% phosphoric acid and 20% acetic acid). To study the effects of surface roughness, a series of these polished disks were exposed to various degrees of surface degradation using abrasive papers of 600, 400, 240 and 60 grit. After surface preparation, the disks were examined using a Sloan-Dektak surface profile tester and a Hommelwerke surface finish tester to determine the rms roughness of each sample.

These samples were then bonded with variable parameters of time and temperature but under a constant pressure of 1800 psi and in an atmosphere of flowing hydrogen gas. Alm [9] determined that bonding temperatures between 1/2 and 2/3 of the absolute melting point of a metal yielded optimum bonding results, which, for copper, implies that a temperature range between 400 and 600°C is needed. Since the diffusion distance in metals is proportional to the square root of time, bonding times of 0.25, 1 and 4 hours were selected so as to double the diffusion distance in each step. As shown later, the optimum bonding conditions were at a temperature of 600°C for one hour, indicating an appropriate selection of bonding times. All surface degraded samples were bonded at 600°C for one hour to provide quantitative comparisons of bond quality between samples with differing initial roughnesses.

Ultrasonic Characterization

The samples were examined using a broadband 2 to 15 MHz focussed transducer. Pulse-echo scans at normal incidence were performed across the diameters of the bonded samples at 30° rotation intervals, with data taken at 0.025 inch increments along each diameter. After scanning, a diamond saw cut was made just above the interface to simulate a perfect reflector to be used as a source for a reference signal with 100% reflection. All time domain signals were Fourier transformed for conversion to their respective frequency spectra; this included deconvolution of the reference signal. The amplitude ratio between a specific signal and the reference signal provided the reflection coefficients as functions of location in the diffusion bond. High reflection coefficients were indicators of poor bonding.

Destructive Test

Several tensile specimens were cut from each diffusion bonded sample with axes perpendicular to the bond line and with final dimensions of 1.0" x 0.25" x 0.10". The tests were performed using an Instron mechanical testing machine at a strain rate of $1.3(10)^{-3}sec^{-1}$. The main parameter obtained from these tests was the ultimate engineering stress of each tensile specimen, which was used as an indicator for the strength of the bond.

Metallographic Evaluation

For metallographic bond line analysis, slices perpendicular to the bond plane of each sample were mounted and polished through Linde B. A light etch was applied in order to characterize the microstructure near the observable bond line. Furthermore, micrographs of the failed diffusion bond surfaces were obtained. From these micrographs, parameters important to diffusion bonding such as the fraction of bonded area and the number of contacts or disbonds per unit area, respectively, were determined. In this analysis, a square grid was superimposed on each micrograph. The fraction of bonded area was then determined by the sum of all line lengths crossing originally bonded areas divided by the total length of lines. A similar analysis was used to determine the average number of disbonds per unit area. This was achieved by counting the number of disbonds in a 3.0" x 3.0" square. After factoring in the magnification, the average number of disbonds per square inch was calculated.

EXPERIMENTAL RESULTS

Results of the ultrasonic characterization are presented in the form of reflection contour maps, such as those shown in Figs. 1 and 2, along with their respective bond line micrographs. These are typical examples of poor (Fig. 1) and good bonding (Fig. 2). In the first case, the reflection coefficients are in excess of 0.1, and the bond line can be clearly seen. In the latter case, the reflection coefficient does not exceed 0.02. The bond line has virtually disappeared due to grain growth across the interface.

The ultimate tensile stresses, obtained from the destructive tests, were then correlated with the ultrasonic reflection coefficient data as shown in Figs. 3 and 4. Figure 3 shows data from diffusion bonds produced over a range of bonding times and temperatures with an original surface roughness of 5.0μin rms. Figure 4 presents similar data from diffusion bonds in which the original surface roughness varied from 5.0μin to 45.2μin rms. It should be noted that in both figures the ultimate stress for a reflection coefficient of R=0 was obtained from a "bulk" specimen (not diffusion bonded) which was heat treated at 600°C for one hour to provide information on the bulk properties of

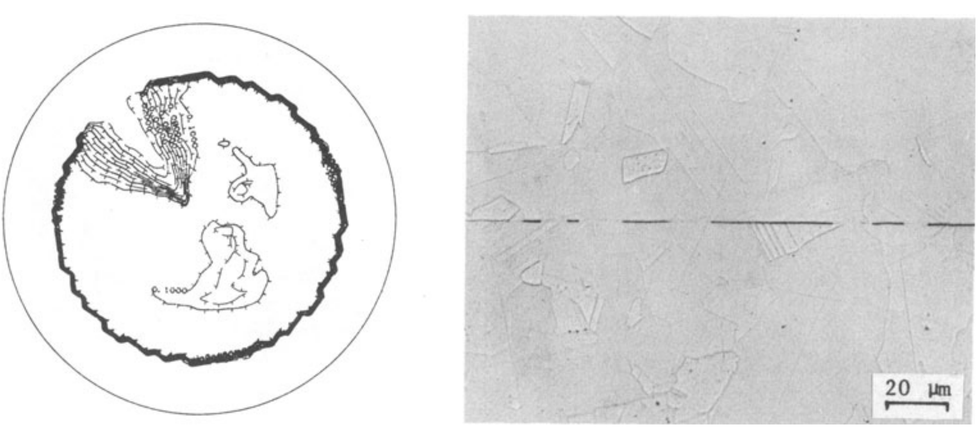

Fig. 1. Reflection contour map (10MHz) and corresponding micrograph for sample bonded at 400°C for 1 hour.

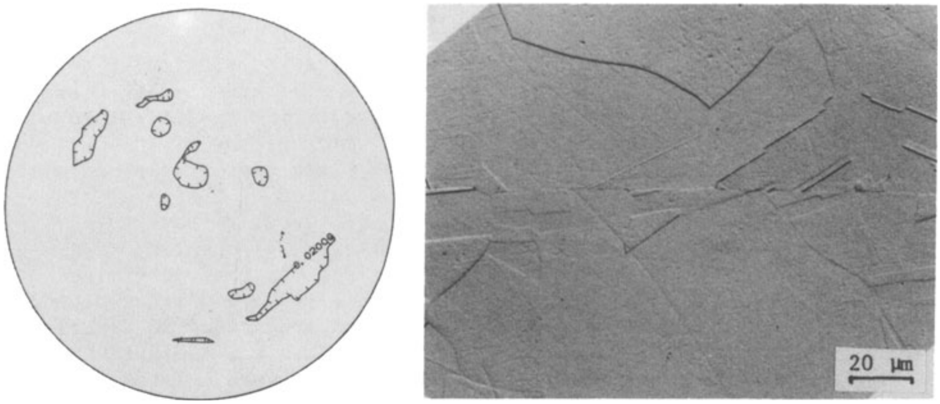

Fig. 2. Reflection contour map (10MHz) and corresponding micrograph for sample bonded at 600°C for 4 hours.

the copper. In both cases, the ultimate stress decreases sharply with increasing reflection coefficient, leveling off at higher reflection coefficients. Comparing Figs. 3 and 4, it becomes apparent that the initial decrease of the ultimate stress is stronger for the originally surface roughened specimens. A possible explanation could be that the originally roughened surfaces promote acoustic scattering, leading to lower values of reflection at the same stress levels.

Interesting to note is the failure mode of the diffusion bonded samples. All diffusion bonded samples failed along the bond lines, except two of four specimens that were bonded at 600°C for four hours. These two specimens failed in the bulk. However, the ultimate stresses achieved in these four specimens were, within experimental error, the same, not showing any trend with failure mode. Examples of fracture surfaces along the bond lines are shown in Fig. 5 for poor, and Fig. 6 for good bonding. Bonding is indicated in both figures by the dark areas, which are actually "dimpled" areas, indicating ductile failure. The bright areas are flat, indicating that no bond had been established. Using such "fractographic" micrographs, the fraction of bonded area, A/A_o, was estimated and correlated with the reflection coefficient R, as shown in Fig. 7. As was expected, R decreases rapidly with increasing A/A_o.

APPLICATIONS TO SPRING MODEL

The above results may now be used to test a "spring model" [6], which is a quasi-static model for the ultrasonic transmission and reflection at imperfect interfaces such as may occur in diffusion bonding. In this model, the interface is represented by a distributed spring, determined by the change in static compliance of the medium with respect to one with a perfect interface. The model has been applied successfully to determinations of fracture mechanics parameters in fatigue crack growth [2] and should, in principle, be applicable to interface problems as may occur in diffusion bonding. At present, we consider such a spring model to be one of the important steps in the development of an overall bond strength model.

The model [6] provides information about the dependence of a "spring constant", κ, on the fractional bonded area. Since the fraction of bonded area is known from the fractographic analysis, κ can be determined. κ is a function of a "normalized" spring constant κ^*, related through

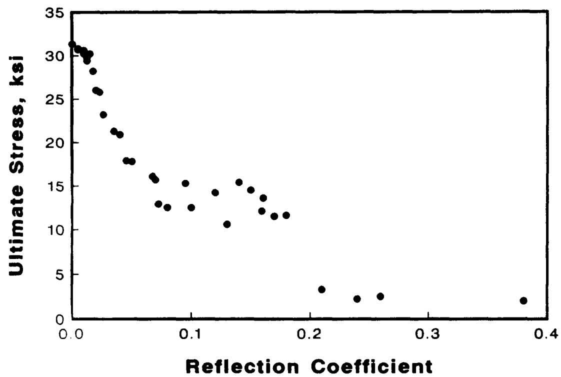

Fig. 3. Ultimate tensile stress vs reflection coefficient (time-temperature variations).

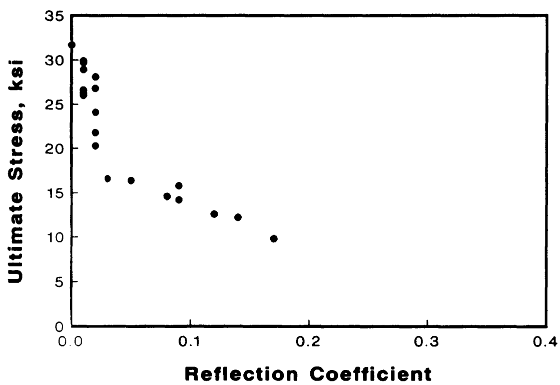

Fig. 4. Ultimate tensile stress vs reflection coefficient (surface roughened samples).

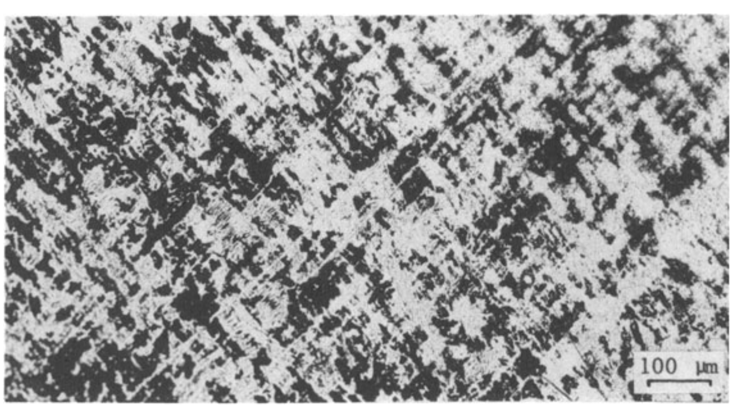

Fig. 5. Fracture surface of sample bonded at 400°C for 1 hour.

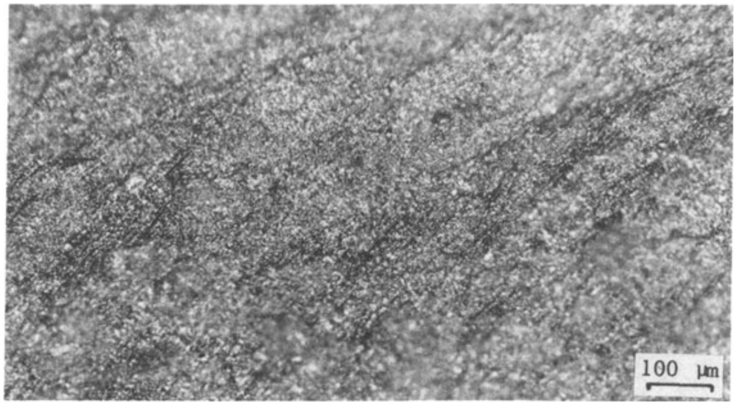

Fig. 6. Fracture surface of sample bonded at 600°C for 4 hours.

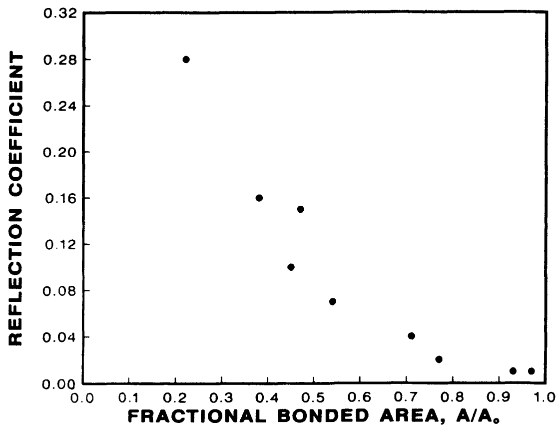

Fig. 7. Reflection coefficient vs fraction of bonded area.

$$\kappa = \kappa^* E/S(1-\nu^2) \qquad (1)$$

where S is the average separation distance between bonded or disbonded areas (whichever is dominant), related to N by:

$$S = (4/\pi N)^{1/2} \qquad (2)$$

E is Young's modulus and ν is Poisson's ratio. κ^* is given in Fig. 8 [6]. The data shown in Fig. 7, then yield a relation between R and κ which is shown in Fig. 9.

On the other hand, this spring model states that the acoustic reflection, R, from an imperfect interface is given by:

$$R = (j\omega z/2\kappa) \,/\, (1+j\omega z/2\kappa) \qquad (3)$$

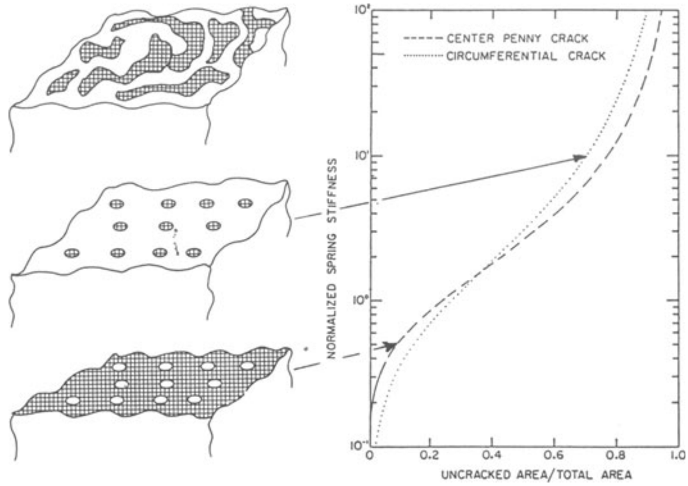

Fig. 8. Determination of normalized spring stiffness.

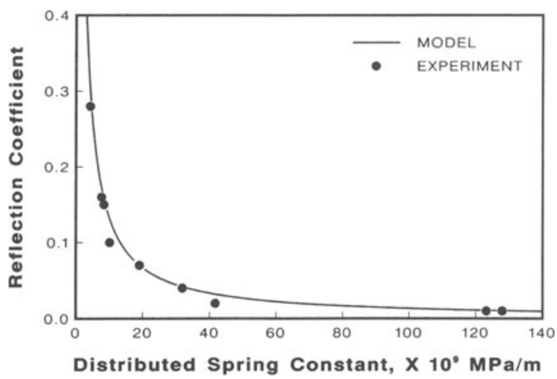

Fig. 9. Reflection coefficient vs interfacial stiffness.

where ω= angular frequency of the acoustic wave and Z = acoustic impedance (density times acoustic velocity in the metal). This equation thus yields a relation between R and κ, which has also been plotted in Fig. 9. For randomly distributed contacts or disbonds, such as shown in Figs. 5 and 6, the experimental results from Eq. (1) are in very good agreement with the theoretical prediction, obtained from Eq. (3).

CONCLUSIONS

A series of diffusion bonds of copper to copper were produced, ultrasonically characterized in reflection and destructively tested, determining the ultimate stress. The best bonds obtained had average ultimate stresses of about 97% of the bulk ultimate stress. In some cases, failure occurred in the bulk. In bonds of lower quality, failure occurs along the bond line and the ultimate stress becomes smaller. When correlating the ultimate stress with the ultrasonic reflection coefficient R, it was determined that σ decreases monotonically with increasing R for a given surface roughness prior to bonding. As this surface roughness increases, with constant bonding parameters (temperature, time and pressure), the reflection coefficient decreases, probably due to additional scattering. Thus it may become necessary to employ a second ultrasonic technique, such as pitch-catch at off-normal incidence or a full characterization of the total diffraction at bond lines. Fractography of the failed bond lines provided information on the fraction of bonded area, A/A_0, the average number of contacts per unit area, N, and the average separation distance between contacts, S. These parameters provide information necessary to test a "distributed spring" model [6]. For randomly distributed contacts or disbonds, respectively, quantitative agreement between experimental results and predictions of the "distributed spring" model has been obtained.

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REFERENCES

1. D. K. Rehbein, J. F. Smith, and D. O. Thompson, in Review of Progress in Quantitative NDE, 3B, D. O. Thompson and D. E. Chimenti, Eds., Plenum Press, NY, 1984, p. 1321.
2. O. Buck, D. K. Rehbein, and R. B. Thompson, in Proceedings of Effects of Load and Thermal Histories of Mechanical Behavior of Materials, TMS-AIME, Metals Park, OH, in press.
3. D. K. Rehbein, D. K. Hsu, R. B. Thompson, and T. A. Jones, in Review of Progress in Quantitative NDE, 6B, D. O. Thompson and D. E. Chimenti, Eds., Plenum Press, NY, 1987, p. 1737.
4. J. A. Regalbuto, Matl. Eval., 30, 3, 1972, p. 66.
5. N. F. Haines, "The Theory of Sound Transmission and Reflection at Contacting Surfaces", Report RD/B/N4711, Central Electricity Generating Board, Research Division, Berkeley Nuclear Laboratories, Berkeley, England, 1980.
6. J-M. Baik and R. B. Thompson, J. Nondest. Eval., 4, 1984, p. 177.
7. Y. C. Angel and J. D. Achenbach, J. Appl. Mech., 52, 1985, p. 33.
8. D. D. Palmer, D. K. Rehbein, J. F. Smith, and O. Buck, in Review of Progress in Quantitative NDE, 6B, D. O. Thompson and D. E. Chimenti, Eds., Plenum Press, NY, 1987, p. 1755.
9. G. V. Alm, Mat. Eng., 70, 1969, p. 24.