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Non-destructive testing and acoustic microscopy of diffusion bonds

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Despite successful applications of diffusion bonding, there is a lack of good quality assessment methods. A survey of published results shows non-destructive testing techniques (NDT) developed for fusion welds to be ineffective in indentifying the much smaller defects found in diffusion bonds. An investigation into the possibilities of using electrical resistance measurements for NDT shows the requirement of equipment with a very high stability and sensitivity. Finally the use of very high frequency ultrasonic sound in an acoustic microscope is considered as a method of defect investigations. This acoustic microscopy proved to be a highly encouraging new line of investigation for the examination of diffusion bonds.

1. Introduction

Diffusion bonding is a high temperature solid-phase joining technique which has been proposed for industrial applications, especially in the field of aerospace. Before widespread use of the technique can occur, large improvements will be needed in the field of quality assurance, by non-destructive testing (NDT). Conventional NDT techniques have been developed for fusion welding and so may not be applicable to solid phase welds.

Most previous studies of diffusion bonds have used destructive techniques to estimate the degree of bonding. Early work assumed that the amount of bonding could be determined by measuring the tensile strength of a bonded sample as a fraction of that of the parent material. This fraction was taken as being the fractional area of the bond [1–3]. This technique has been examined critically [4] and was found to be inaccurate because a bond can display the tensile strength of the parent material when as little as 80% bonding occurs. However, such poorly bonded specimens may have poor fracture toughness properties and, therefore, tensile testing does not give a good guide to satisfactory bonding. The other destructive method used is of sectioning the bond the assessing the bonded area metallographically [5]. This seems to give good results, albeit with a certain degree of scatter. All destructive tests, although they will be useful in the development stage of the process, cannot be used to routinely examine finished components.

For quality control in fusion welds, several non-destructive techniques are used. Some of these NDT techniques have been tried with diffusion bonds and an analysis of their potential is presented with respect to the defect size in diffusion bonds. As a development of ultrasonic NDT, a study has

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been made as to the applicability of acoustic microscopy to examining diffusion bonds. The technique is similar to ultrasonic crack detection but operates at much higher frequencies and uses focused beams [6]. Acoustic microscopy is still in the development stage and, in transmission, suffers resolution problems due to the scattering effects of microstructure. However, preliminary results are encouraging. It is hoped that the technique might eventually lead to the development of better ultrasonic NDT methods for characterizing solid-phase welds.

2. Surface roughness induced defects in diffusion bonds

One major difference between fusion welds and solid phase welds is the defect size in incomplete bonds. In fusion welds defects are formed, for example, by hot tearing and incomplete weld penetration. The resulting defects, although few in number, are usually large in size (on the scale of millimetres). In diffusion bonding and some other solid-phase techniques, the size of defects is determined principally by the scale of roughness of the surfaces being bonded [7]. The difference in scale is so great that the techniques developed for flaw assessment in fusion welds are not sensitive enough to detect the much smaller defects in diffusion bonds.

2.1. Surface roughness prior to diffusion bonding

When diffusion bonding, the two surfaces to be joined are usually machined flat and parallel prior to bonding. The degree of residual surface roughness is clearly dependent on the material and machining technique. The techniques most commonly used are off-centre lathing and grinding. Previous work of the authors [7] implied that the finer the roughness of the surface prior to bonding, the easier the bonding will be. Therefore, in commercial applications, very fine scale roughnesses are to be expected, giving corresponding small defect sizes.

Fig. 1 shows roughness traces of surfaces prepared for bonding by off-centre lathing and grinding. Both processes give a major roughness wavelength of about 50 μm. The lathing process gives a roughness height of about 2 μm while the grinding gives about 0.5 μm. Grinding gives a very irregular surface which may induce any interface voids to break up into smaller ones (see Fig. 5). Although much smoother surfaces can be produced (e.g. by diamond lapping), it is believed that in industrial applications roughnesses prior to bonding will be of magnitudes similar to those shown in Fig. 1 and these are perfectly feasible for satisfactory bonding.

In diffusion bonding the mechanism of bond formation is believed to be the deformation of the surface roughness, to allow intimate metal–metal contact followed by bonding, which is achieved by diffusional and creep processes [7]. Initially, the two surfaces are brought together with their grinding marks parallel so creating long, cylindrical voids. The process can also be thought of as the reduction in volume and elimination of an array of these voids on the bond plane, and so an incomplete bond can be thought of as a planar defect in the material, the defect consisting of an array of long narrow voids of a maximum cross-section determined by the initial roughness. This array of voids is believed to cause a much reduced fracture toughness and its presence would be generally deleterious to performance.

2.2. Defect size in incomplete diffusion bonds

The bond interface voids or defects are found to be of the same order of size as the initial roughness. Fig. 2 shows a bond made from a copper surface lathed flat to the degree of roughness shown in Fig. 1a. Although these voids are quite large, investigation using a scanning electron microscope (SEM) reveals more, smaller voids (Fig. 3). The somewhat finer surface produced by grinding (Fig. 1b) produces a much finer scale void distribution at a similar stage in the bonding

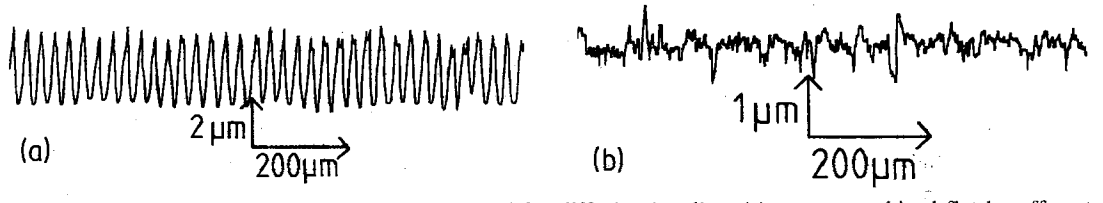

Figure 1 Surface roughness traces of surfaces prepared for diffusion bonding; (a) copper machined flat by off-centre lathing, (b) iron ground flat by 6A K8 V75 grinding wheel.

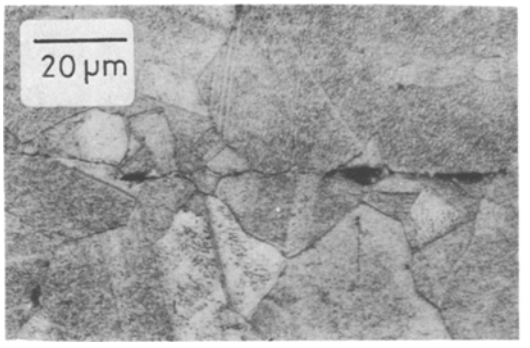

Figure 2 Optical micrograph of voids in a copper diffusion bond. Bonding conditions were 120 min at 550° C with 35 MPa pressure; the surface was prepared as in Fig. 1a.

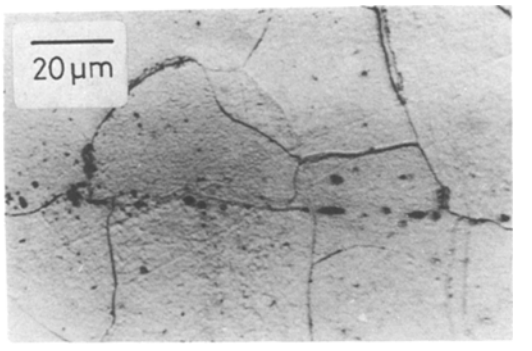

Figure 4 Optical micrograph of voids in an iron diffusion bond. Bonding conditions were 10 min at 1000° C with 7 MPa pressure; the surface was prepared as in Fig. 1b.

process (Fig. 4). Again SEM studies show there to be very small voids present (Fig. 5).

Both these example bonds have a fractionally bonded area of about 0.7 (determined metallographically). Therefore, it can be seen that, even in comparatively poor diffusion bonds, the defect size is very small and is on the scale of microns. In comparison, fusion welds have much fewer defects but of a size many orders of magnitude greater.

3. The non-destructive testing of diffusion bonds

NDT has been used as a quality control technique in fusion welding for many years. If diffusion bonding is to find widespread industrial use, some accurate form of NDT will be needed. Conventional NDT techniques in fusion welding are X- and gamma-radiography and ultrasonic testing. It is uncertain as to whether these techniques, adequate for detecting millimetre sized flaws, will detect the micron sized voids in a poor diffusion bond. Attempts have been made to use electrical resistivity measurements on diffusion bonds as an NDT technique and this is examined critically.

3.1. Radiography

Radiographic examination can be thought of as casting a shadow of the joint; any voids present will result in less absorption of the X- or gamma-rays and so will show up on the detector film. As the extent of magnification from such a film is limited, this technique appears to be on too coarse a scale for diffusion bonding. Two previous studies of NDT on diffusion bonds have investigated the possibilities of using, and the effectiveness of, X-ray techniques [8, 9]. Both studies concluded that such radiographic techniques did not pick up interface voids unless they were of unrealistically large sizes.

3.2. Bond resistivity measurements

A potential NDT technique for diffusion bonds is the measurement of bond resistivity. The conductivity of a piece of metal is proportional to the area through which electrons can migrate. If there is a partial bond in the material, this migrational area will be reduced in the region of the bond and thus the resistance of the material will be greater than that of a similarly sized perfect specimen.

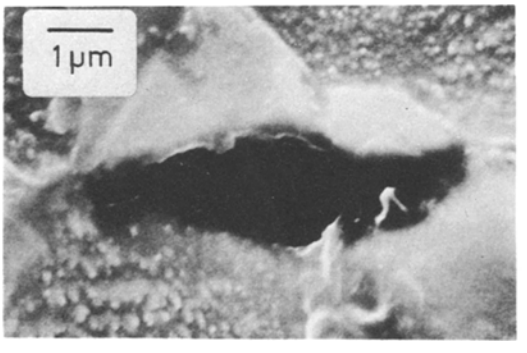

Figure 3 SEM micrograph of the copper bond in Fig. 2.

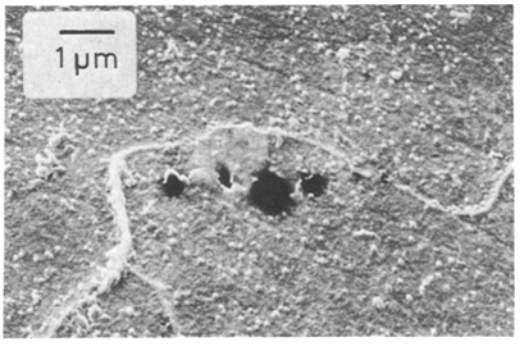

Figure 5 SEM micrograph of the iron bond in Fig. 4.

The standard technique for measuring the resistance of such a bond is the "four contact method". A large constant current is passed down the specimen being tested by two power contacts. The potential difference between points on the specimen surface is measured by two other contacts. The voltage drop along a length of specimen containing the bond can be compared with that of an identical length of perfect material and there should be a simple relationship between the difference in conductivity and the total bonded area. In practice, the measuring contacts will need to be some distance either side of the bond to ensure that they are in the region of planar equipotential surfaces. This and other practical considerations, suggests that the measuring contacts can be no nearer to each other than one millimetre.

In order to estimate the required equipment sensitivity for testing diffusion bonds, the difference in potential drop across a poor bond and a perfect bond may be calculated. If the measuring contacts are placed a distance $l/2$ either side of a bond of nominal area A_1 in a material of resistivity ρ, then the measured resistance is;

$$R = \frac{\rho l}{A_1} \tag{1}$$

However, when voids are present because of incomplete bonding, an extra resistance term, ΔR, occurs. For the case of an isolated planar array of long thin voids, this term has been derived by conformal mapping [10]. If the true bonded area is A_2 and the spacing between the void centres, which is equivalent to the mean wavelength of prior surface roughness, is $2h$, then the additional resistance is:

$$\Delta R = \frac{4h\rho}{\pi A_1} \ln \frac{a^2+1}{2a}$$

where

$$a = \cot \frac{\pi}{2} \frac{A_2}{A_1} + \operatorname{cosec} \frac{\pi}{2} \frac{A_2}{A_1} \tag{2}$$

For a given specimen, the resistivity, current and specimen size are constant. Therefore the potential difference between the measuring contacts is:

$$V_B = \rho I \left[\frac{l}{A_1} + \frac{4h}{\pi A_1} \ln \left(\frac{a^2+1}{2a} \right) \right]. \tag{3}$$

By differentiating Equation 3, an estimate can be made as to how a change in bonded area will affect the measured potential.

$$\delta V_B = - \frac{2\rho I h (a^2-1)}{A_1^2 (a^2+1) \sin \frac{\pi}{2} \frac{A_2}{A_1}} \delta A_2. \tag{4}$$

The instrument sensitivity required is determined by a ratio $\delta V_B / V_B$ as a function of the fractional change in bonded area $\delta A_2 / A_1$. The variation of this sensitivity with total bonded area is indicated in Fig. 6 for a bond of surface roughness wavelength ($2h$) 50 μm and a contact separation (l) of 1 mm. As can be seen, the early stages of bonding can be monitored quite easily with potential drop equipment. However, above about 70% bonding, the required sensitivity increases indefinitely. This conclusion might be modified when the assumption about void geometry (namely, that their height is small compared with their width) ceases to be valid. Nevertheless, for the latter stages of bonding, highly accurate equipment will be needed both for measurement of potential difference and for ensuring a stable constant current.

An attempt has been made by the authors to verify nominally good diffusion bonds with equipment normally used for crack monitoring in fatigue experiments. The results were inconclusive as the

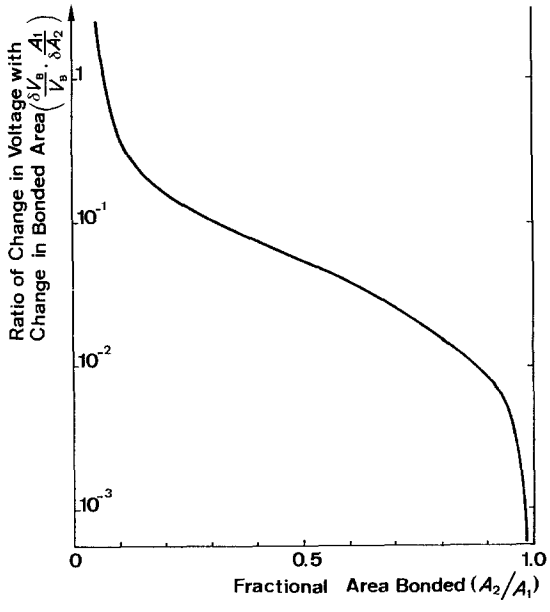

Figure 6 The variation in required e.m.f. measurement sensitivity as bonding proceeds. The sensitivity is measured in terms of the fractional change in voltage across the bond with fractional change in bonded area at a given stage of bonding. Contact separation of 1 mm (l), small void height (1 μm), average void separation of 50 μm ($2h$) and a specimen area of 10^{-4} m^2 (A_1).

equipment did not have the required stability or sensitivity. A serious problem with the technique is that the changes being observed are so small that they may be swamped by minute variations in experimental conditions or material properties. Nevertheless, some workers have reported a certain amount of success using the technique [11–13]. However, this was only at very early stages of bonding when the actual bonded area is very small and the potential difference correspondingly large.

The technique needs more consideration, possibly some development in equipment design could make a large difference. One suggestion is to use four measuring electrodes, two measuring the parent material and two the bond. A system of differential amplification could then be used for greater sensitivity.

3.3. Ultrasonic testing

With this technique, ultrasonic vibrations are transmitted through the material being tested. When these vibrations intersect a discontinuity, e.g. a void, they are reflected and so the discontinuity detected. The technique is used for the assessment of fusion welds and is well understood. Unfortunately, the ultrasonic vibrations used have a frequency in the range of tens of MHz. The speed of sound in metals is typically about 5000 $msec^{-1}$, which gives a wavelength in the range of about 100 to 200 μm for the ultrasound. This wavelength is considerably larger than the defects expected in the latter stages of diffusion bonding and so the defects would not be detected. Ushakova *et al.* [8] confirmed this experimentally and reported ultrasonic NDT to be of no use for diffusion bonds. Rehder and Lovell [9], however, give a guarded recommendation for the technique although the bonds used in their investigation had artificially induced voids of about 20 μm × 200 μm, much larger than the defect size implied in Section 2 of this work.

4. Acoustic microscopy of diffusion bonds

If the wavelength of the ultrasound used to inspect bonds is much larger than the size of unbonded areas, then the amount of reflected power, or alternatively the reduction in transmitted power, depends on the number and size of the defects. In conventional non-destructive ultrasonic inspection, plane waves are used in order to determine these parameters. In the acoustic microscope, the ultrasonic beam (which may be of a much shorter wavelength) is focused to a spot whose size is limited chiefly by diffraction to about one wavelength, and a scanned image is then built up. Sintered bonds between a carbon diamond compound and a steel base have previously been examined in a scanning acoustic microscope operating at 12 MHz with a resolution of better than 1 mm [14]. At these frequencies, it was possible to obtain good images indicating the variation in bond quality over the interface.

In order to study the details of their developments, diffusion bonds have now been imaged in a scanning transmission acoustic microscope operating at 140 MHz. This gives a resolution within the specimen of approximately 50 μm, though defects smaller than this may still be detected.

Three specimens of 10 μm grain size En8 steel were chosen as follows:

(a) a single piece of material with no bond;

(b) two pieces bonded together with a pressure of 20 MPa at 900° C for 3 min (anticipated 70% bonded area);

(c) two pieces bonded under the same conditions as (b) but for 20 min (anticipated 100% bonded area).

The specimens were then ground and polished to a thickness of 0.9 mm and a 1 μm surface finish with the faces flat and parallel. For specimens (b) and (c), the bond was midway between the faces.

An image of the unbonded specimen (a) is shown in Fig. 7. It can be seen that the dominant feature of the image is its dappled appearance which has almost 100% contrast. The origin of this contrast is believed to lie in multiple scattering at grain boundaries. It is a considerable nuisance as far as the imaging of diffusion bonds is concerned and various possible ways of reducing it are being considered. These include using alloys with a lower crystal elastic anisotropy, using a much smaller grain size, or simply by preparing much thinner specimens. (En8 may suffer more from this problem of scattering because of the fine two phase structure present.) The effect has a very strong frequency dependence and it can be almost eliminated by moving to lower frequencies. However, such lower frequencies also eliminate the contrast from the bond itself, as shown in Fig. 8 which is an image of the poor bond (b) at 45 MHz (cf. Figs. 9 and 10). Therefore, in the present investigation, it was decided to use 140 MHz despite the unwanted dappled effect.

Figure 7 Scanning acoustic microscope (SAM) image of En8 steel (a) at 140 MHz. Dappled contrast is believed to be caused by scattering from grain boundaries.

The bond (b) made for 3 min is a rather poor one. The bonded interface is expected to contain long narrow voids associated with the grinding of the surface prior to bonding and such voids would appear dark in transmission. An image of bond (b) at 140 MHz is shown in Fig. 9; comparison with Fig. 7 shows that superimposed on the dappled effect there are features that run approximately vertically. To confirm that this was not an artefact of the imaging system, the specimen was rotated anticlockwise through 45° and imaged again, this is shown in Fig. 10. It is believed that the dark regions running across Figs. 9 and 10 correspond to voids in the diffusion bond. The image of a 100 mesh grid (spacing 254 μm) is shown in Fig. 11; this indicates the scaling in the other figures and shows the distortion due to temporary scanning electronics.

Finally, the relatively good bond (c) was imaged and the result is shown in Fig. 12. No features appear in this picture which can be distinguished from the dappling. Therefore, the imaging of smaller residual voids in good diffusion bonds must await the development of techniques for reducing the effect of grain boundary scattering. Nevertheless, it is encouraging to be able to distinguish between good and bad bonds.

5. Conclusions

When considering developing a non-destructive testing technique, it is necessary to know the maximum size and distribution of defects that would not adversely affect the properties for given service conditions. Once these have been determined, the required accuracy and resolution of any potential NDT technique can be defined.

Figure 8 SAM image of diffusion bond (b) taken at 45 MHz. Dappled pattern disappears as does most of the contrast from the image. The image contrast has been enhanced photographically, in reality there is almost no contrast visible.

Figure 9 SAM image of diffusion bond (b) taken at 140 MHz. Several black vertical markings are visible, possibly caused by cylindrical voids on the bond interface.

Current conventional NDT techniques developed for inspecting fusion welds can detect excessively large flaws in diffusion bonds (for instance, those caused by gross errors in surface preparation or bonding conditions or by massive contamination of the parts being bonded). However, it has been shown by previous workers that most of the conventional NDT techniques cannot be used to detect the very fine size defects associated with diffusion bonds, and these fine defects are known to adversely affect properties.

One possible new NDT technique, bond resistivity measurement, has been subjected to a brief analysis. This indicated that equipment of very high stability and sensitivity would be needed to investigate the latter stages of diffusion bonding. Although some work has been done in this area, it really requires an intensive study to determine its feasibility. The idea of differential amplification needs perhaps to be explored. The ultimate target of such resistivity techniques would lie in the direct monitoring of bonding *in situ*. The idea of having an "extent of bonding" measurement device on future bonding apparatus would be most attractive.

Finally the acoustic microscope, this is very much a new technique and problems must be expected as it is still in the development stage. The main problem encountered is the severe information loss caused by image dappling at higher frequencies. It is possible that En8 steel was a poor choice of material for an initial study of diffusion bonds by acoustic microscopy. Work is currently in progress using bonds fabricated in nominally

Figure 10 SAM image of diffusion bond (b) taken at 140 MHz. The specimen is rotated 45° from its alignment in Fig. 9, the vertical markings have also rotated.

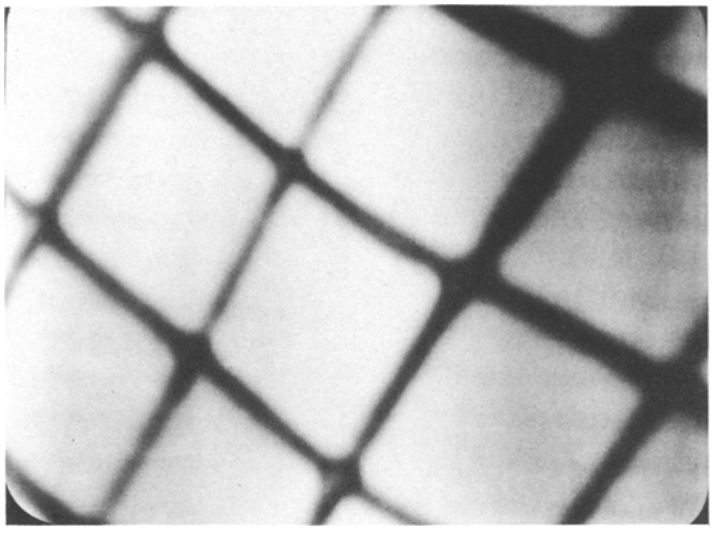

Figure 11 SAM image of a 100 mesh grid with a nominal spacing of 254 μm. This is to the same scale as the other SAM images.

pure iron with different grain sizes. Materials with a lower elastic anisotropy (e.g. tungsten) could also be used in development studies (although difficult to diffusion bond) but ultimately the dappling problem will need to be resolved.

It must be emphasized however, that even at this early stage acoustic microscopy appears to be second only to sectioning the bond in terms of void resolution. Of course, the former too is, at the moment, a destructive technique but developments in reflection acoustic microscopy may eventually enable the examination of bulk diffusion bonds. Possibly, experience in the use of these high frequency ultrasonic techniques may lead to the design of NDT equipment suitable for diffusion bonds.

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Figure 12 SAM image of diffusion bond (c) taken at 140 MHz. No discernable features other than the dappled pattern are observed.

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