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Impact strength of En8 steel diffusion bonds

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The use of diffusion bonding for the joining of steel is not widespread due partly to the large number of alternative welding processes and partly to a lack of information on mechanical properties of the resultant bonds. In this paper, data are provided on both tensile and impact properties of diffusion bonds in a plain carbon steel containing 0.4 % C. Several hypotheses are proposed for the observed low impact properties and one of these, the presence of a planar layer of ferrite at the bond line, is discounted.

1. Introduction

Diffusion bonding is a solid-state joining process whereby the joining of two contacting surfaces is achieved on a microscale by diffusion-controlled processes with negligible macroscopic deformation. The occurrence of the diffusion-controlled processes is induced by the application of heat and pressure for a finite time. The majority of applications for diffusion bonding have been found in high-technology industries, e.g. in the aerospace industry for the fabrication of titanium alloy structures [1]. The use of diffusion bonding for the joining of steel is not widespread, due partly to the large number of alternative welding processes and partly to the lack of information on the mechanical properties of the resulting bonds.

In previous work [2], it has been shown that diffusion bonds can achieve parent metal tensile strengths although the bonded interface may contain up to 20% unbonded area. However, for plain carbon steels, Charpy impact testing appears to be a more critical test of the quality of diffusion bonds which, in general, have been found to have poor impact properties [3, 4]. These low impact properties may be attributed to the presence of one or more of the following:

(1) a planar layer of ferrite at the bonding interface;

(2) voids and/or inclusions at the bond interface;

(3) contaminant and/or segregants at the bond interface.

One aim of the work described here is to provide data on the mechanical properties, especially impact properties, of diffusion bonds in a plain carbon steel and to relate these properties to the observed bond microstructures. A second aim is to examine whether the bond microstructures can be used to confirm one of the above hypotheses postulated for low impact strengths.

2. Experimental procedure

2.1. Material

The material chosen for the investigation was a commercially available cold-drawn plain carbon steel containing approximately 0.4 wt% C (16 mm diameter En8). Analyses of two separate batches of steel used (designated En8 and En8-A) are shown in Table I. This steel was chosen because of its wide industrial applications and also because it has been the subject of previous diffusion-bonding studies [3, 5]. Prior to use, the steel was given a stress-relieving heat treatment in a vacuum furnace for 1 h at 550° C followed by an air cool. This was carried out in order to minimize the residual stresses which can cause the splitting open of the diffusion-bonded interface after bonding has been completed [2].

2.2. Specimen preparation and bonding

The En8 steel rod was cut into 55 mm lengths and one of the end faces of each resulting piece was hand-ground in a jig on water-lubricated silicon carbide paper. Each piece was finish-ground on 1200 grit paper which resulted in parallel grinding marks on the surface. Thus the surfaces to be

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TABLE I Compositions of materials used (wt %)

Material	C	S	P	Si	Mn	Ni	Cr	Al	Nb	Sn	Fe
En8	0.38	0.037	0.012	0.29	0.83	0.16	0.09	0.031	< 0.005	0.02	balance
En8-A	0.43	0.032	0.007	0.24	0.84	0.18	0.10	0.040	< 0.005	0.02	balance

joined were perpendicular to the length of the specimen and had a surface roughness whose peak-to-valley height was better than 0.4 μm and a wavelength of 40 to 50 μm (both measured on a Surfcom surface profilometer). The surfaces were degreased with acetone prior to joining and were bonded with the grinding marks on the contacting surfaces aligned parallel to each other.

All bonds were fabricated in a small diffusion bonding unit consisting of a vacuum chamber (10^{-2} to 10^{-3} Pa), radio frequency heating source and a motor-driven ram to apply the load. Further details of the diffusion bonding unit have been provided elsewhere [6]. For each bond, the two pieces of steel to be joined were held vertically between two rams. The load was applied to the specimen by an electric motor driving the top ram, and an induction heating coil was used to provide a hot zone which was centred on the region in which the surfaces to be joined were in contact. The temperature at the bond zone was measured using a chromel/alumel thermocouple spot welded to the specimen close to the contacting surfaces.

The pieces to be joined were placed in the diffusion bonding apparatus under zero load and the chamber pumped down to a vacuum of 10^{-2} Pa or better. Before the bonding load of 20 MN m^{-2} was applied, the temperature was raised to that required for bonding, i.e. 800, 900, 1000 or 1060° C. After a bonding time of 20 min had elapsed, the radio frequency heating source was switched off and the load removed. The specimens were gas quenched using argon and allowed to cool to room temperature. In order to compare the mechanical properties of the bonds with those of the parent metal, control specimens (110 mm in length) were subjected, in the bonder, to a heat and pressure cycle identical to that given to the diffusion-bonded specimens.

Some of the specimens which had been diffusion bonded at 900° C were given the following post-bond heat treatments:

(1) post-bond thermal cycle. Whilst still in the diffusion bonding unit, the bonded specimen was cooled to 600° C, reheated to 900° C for 30 sec and then cooled to room temperature. This thermal cycle was performed in order to remove the planar layer of ferrite at the bond interface (see Section 3.1);

(2) post-bond annealing. The bonded specimen was allowed to cool to room temperature before being given one of the following heat treatments in a vacuum furnace: (a) 1 h at 600° C; (b) 1 h at 900° C; (c) 24 h at 900° C.

These heat treatments were used in order to reduce the size and density of the bond line voids (see Section 3.1).

2.3. Bond assessment

2.3.1. Optical and scanning electron microscopy

Sections of diffusion bonds, cut perpendicular to the direction of the original grinding marks, were mounted for optical microscopy so that the joint zone could be examined. The specimens were polished to a 1 μm diamond finish and then etched in 2% Nital.

An ISI 100A scanning electron microscope was used to examine the fracture surfaces of the tensile and impact specimens. The microscope was also used to examine the polished and etched metallographic cross-sections of the bonds in order to assess the density and sizes of the interfacial voids. The mean size of the bond line voids was assessed quantitatively by taking measurements from micrographs of the load line voids. The density of the bond line voids was assessed qualitatively.

2.3.2. Tensile testing

Standard Hounsfield no. 16 tensile specimens were machined from diffusion bonds so that the bond line was positioned in the centre of the resulting tensile specimen. Tensile tests were also performed on parent metal control specimens which had been subjected to a bonding cycle. The specimens were tested using a 100 kN Instron machine at the Welding Institute. The yield stress, nominal UTS and reduction in area at failure were all recorded.

2.3.3. Impact testing

Standard Charpy impact specimens (10 mm x 10 mm x 60 mm) were machined so that the bond

line was positioned in the centre of the specimen and so that the direction of the original grinding marks was parallel to that of one of the sides. A standard 2 mm deep notch was machined along the bond line so that it was positioned perpendicular to the direction of the original grinding marks. Parent metal control specimens were machined in an identical manner. Initially, the specimens were tested at room temperature which is within the ductile brittle transition temperature range for En8 steel. Consequently, some parent metal controls failed in a ductile manner while others (with larger grain size) failed in a brittle manner. Accordingly, the test temperature was raised to + 80° C and this ensured that the parent metal control specimens consistently failed in a ductile manner and so facilitated comparisons of impact strengths between the parent metal control specimens and diffusion bonds.

3. Results and interpretation

3.1. Metallographic examination of bonded interfaces

3.1.1. Optical microscopy

Optical microscopy showed that the bond line for diffusion bonds fabricated at 800° C is planar with both ferrite and pearlite grains along the boundary. For bonds fabricated at 900 and 1000° C, each bond line was delineated by a planar layer of ferrite and little grain growth occurred across it (Fig. 1a). Adjacent to the bond line, it was apparent that only moderate grain growth of the austenite phase had taken place during bonding, the prior austenite grain boundaries being delineated by ferrite. In contrast, at a bonding temperature of 1060° C, grain growth across the boundary was observed. Thus the planar austenite grain boundary at the bond line has migrated during bonding so that only a few regions of the joint interface were marked by ferrite (Fig. 1b).

The effect of either a post-bond thermal cycle or a post-bond anneal at 900° C on specimens bonded at 900° C was to remove the planar layer of ferrite present at the bond line (Fig. 1c). The planar layer of ferrite was not removed by a post-bond anneal at 600° C which is below the austenite temperature. Clearly, on reheating a bonded specimen to 900° C, austenite grains re-nucleate and grow across the original bond interface and so a planar boundary in the austenite is not formed. Hence, on subsequent cooling, the bond line will no longer be delineated by a planar ferrite boundary. The bond lines in specimens which received post-bond thermal treatments at 900° C were still delineated by remaining bond line voids, the size and density of which were assessed in a manner described below.

3.1.2. Scanning electron microscopy

Scanning electron microscopy was used to examine the size and density of bond line voids. It was apparent that voids always remained after bonding was completed and, from Table II, it appears that the void size varies little with increasing bonding temperature. In contrast, the void density was observed to decrease with increasing bonding temperature. With regard to post-bond annealing, while the void size decreases only slightly with increasing "severity" of the post-bond treatment, the void density decreases considerably. The post-bond annealing treatments allow grain boundary and volume diffusion to close bond line voids and thus result in a reduction in void density. This is consistent with a proposed model for diffusion bonding [7] in which it is suggested that both the size and density of isolated voids can be reduced by diffusion mechanisms in the final stages of bonding.

As has been described, the bond lines of the specimens bonded at 900° C were delineated

TABLE II Average dimensions of bond line voids

Material	No. of voids measured	Bonding temperature (° C)	Post-bond thermal cycle	Void width (μm)	Void height (μm)
En8	44	800	–	2.1 ± 1.4	1.6 ± 0.6
	42	900	–	3.2 ± 1.4	2.0 ± 0.9
	31	1000	–	3.1 ± 1.9	2.5 ± 1.5
	22	1060	–	4.1 ± 3.3	1.6 ± 0.6
En8-A	77	900	–	1.8 ± 1.1	1.3 ± 0.6
	46	900	Thermal cycle	1.6 ± 1.3	1.0 ± 1.0
	46	900	1 h at 600° C	1.0 ± 0.8	0.8 ± 0.5
	50	900	1 h at 900° C	0.9 ± 0.5	0.6 ± 0.4
	51	900	24 h at 900° C	1.4 ± 0.9	0.9 ± 0.3

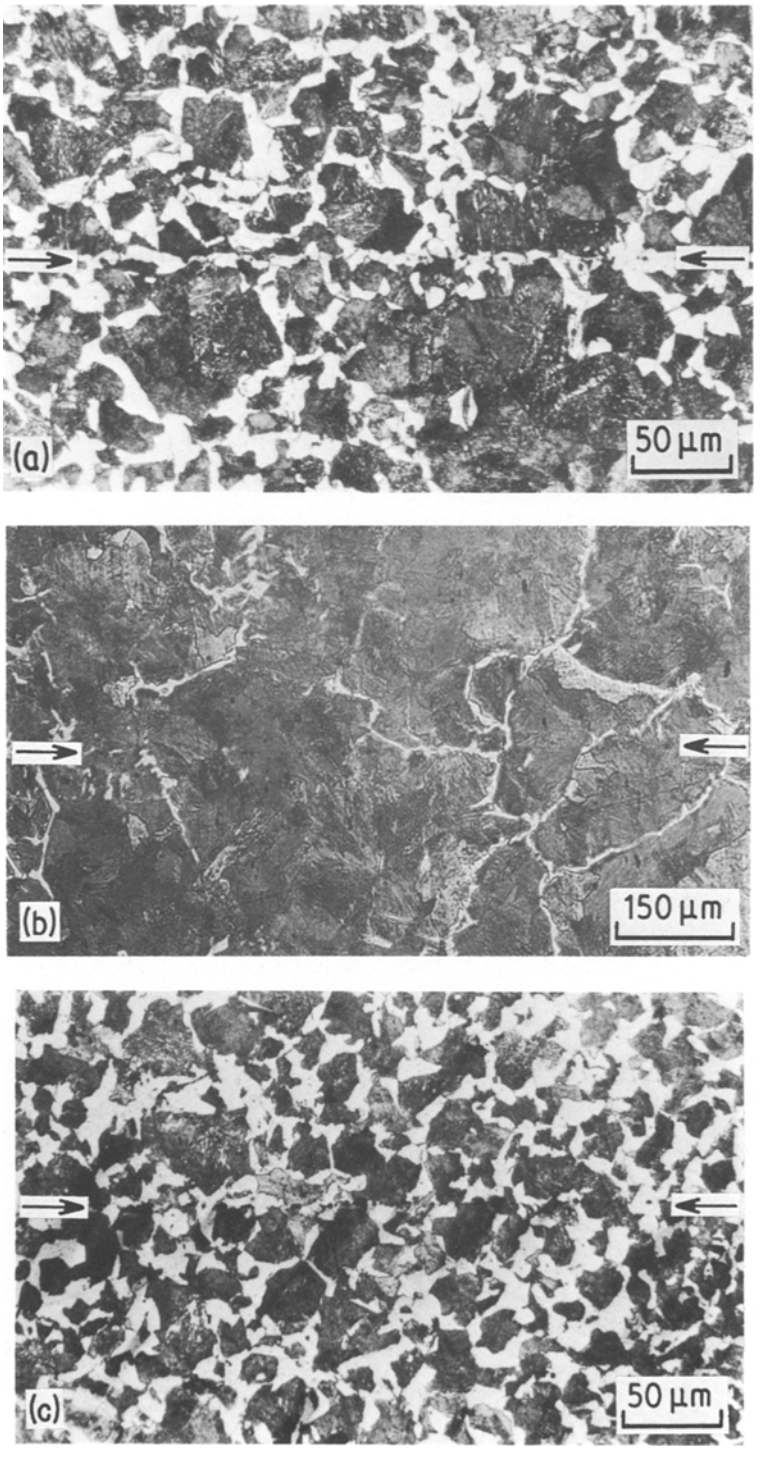

Figure 1 Optical micrographs (bond lines arrowed) of sections through En8 bonded for 20 min and 20 MN m^{-2} at temperatures of: (a) 900° C; (b) 1060° C; (c) 900° C with post-bond thermal cycle.

mainly by ferrite although a few pearlite colonies were seen to grow across some interfaces. Thus interfacial voids occur mainly in the ferrite or at ferrite/pearlite boundaries although a few voids were visible in pearlite colonies (Fig. 2a). The bond lines of bonds fabricated at 1000° C were similar to those of bonds fabricated at 900° C except that at 1000° C more grain growth

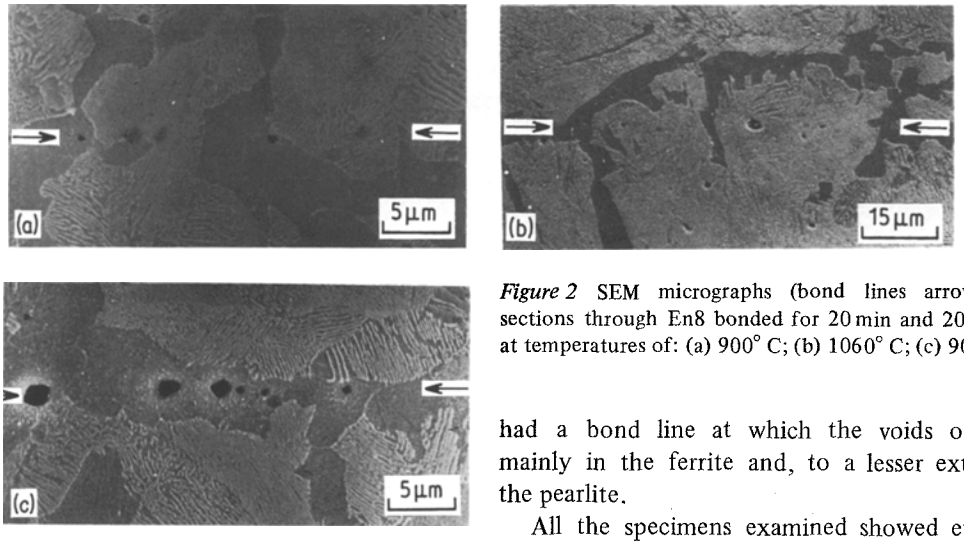

Figure 2 SEM micrographs (bond lines arrowed) of sections through En8 bonded for 20 min and 20 MN m^{-2} at temperatures of: (a) 900° C; (b) 1060° C; (c) 900° C.

occurred across the bond lines and consequently more voids were visible in pearlite colonies. For bonds fabricated at 1060° C (Fig. 2b), massive grain growth across the bond lines has taken place and the bond line voids occur mainly in pearlite colonies. As the bonding temperature increased, the volume fraction of pearlite in the resultant microstructure was seen to increase to above that for the equilibrium microstructure; this is a consequence of the higher cooling rates.

The bond interfaces in specimens which had received a post-bond thermal cycle at 900° C were no longer delineated by planar layers of ferrite and so bond line voids were visible in both regions of ferrite and pearlite. Correspondingly, specimens which had received a post-bond anneal at 600° C had a bond line at which the voids occurred mainly in the ferrite and, to a lesser extent, in the pearlite.

All the specimens examined showed evidence of both void packets as well as isolated voids. Void packet formation occurs when a void which has retained its secondary wavelength roughness shuts to form a packet of voids (Fig. 2c). The formation of void packets has been suggested in theoretical considerations of diffusion bonding by Derby [7] and Allen and White [8].

3.2. Tensile tests

The variation of the tensile properties of En8 steel diffusion bonds with bonding temperature is shown in Fig. 3. The nominal UTS, yield stress and reduction in area values perhaps increase very slightly with increasing bonding temperature but the increases are not considered to be significant. The values of the tensile properties of the diffusion-bonded specimens are very similar to those of the

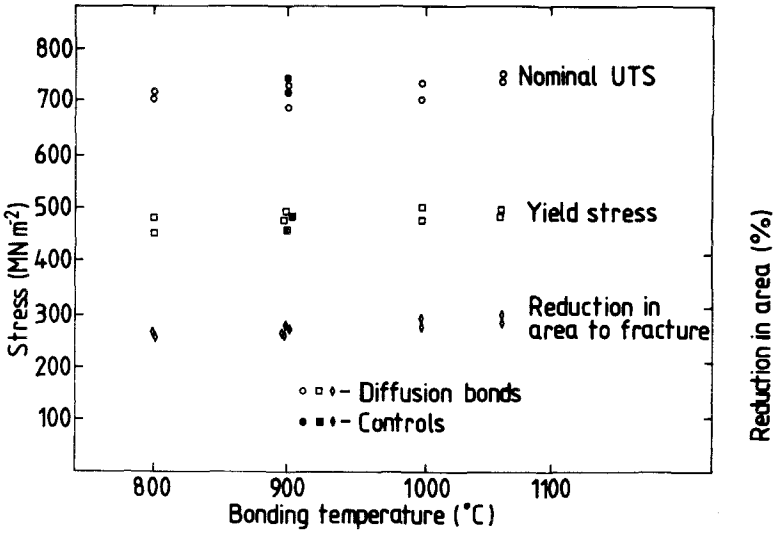

Figure 3 Variation of tensile properties with bonding temperature (diffusion bonds in En8 with a bonding time of 20 min at a pressure of 20 MN m^{-2}).

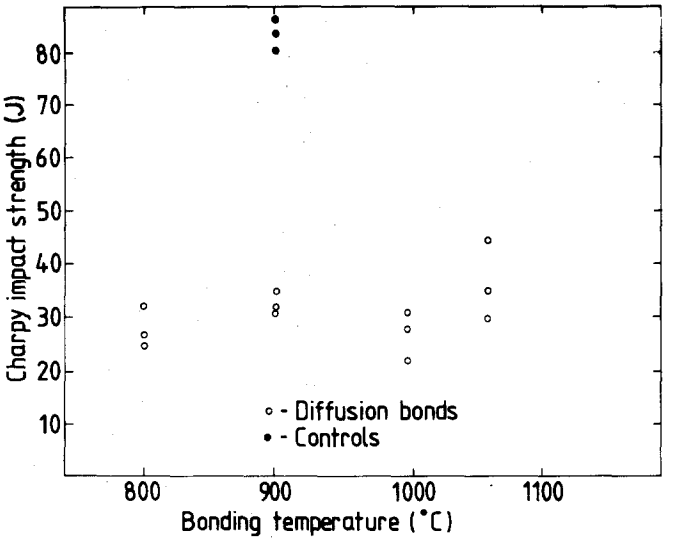

Figure 4 Variation of impact strength with bonding temperature (diffusion bonds in En8 with a bonding time of 20 min at a pressure of 20 MN m^{-2}, impact tested at room temperature).

unbonded control specimens, which is not surprising since all specimens tested failed by necking in the parent metal away from the bond line.

3.3. Impact tests

3.3.1. Variation of impact strength with bonding temperature

The variation of impact strength with bonding temperature of bonds fabricated in En8 and tested at room temperature is shown in Fig. 4. It can be seen that, despite the scatter, the impact strengths of the diffusion-bonded specimens vary little with bonding temperature and in all cases are significantly less than those of the control specimens. The variation in impact strength of bonds fabricated in En8-A and tested at + 80° C is shown in Fig. 5. The impact strengths of the parent metal controls decrease with increasing bonding temperature while those of the bonded specimens reach a maximum at the bonding temperature of 900° C. In order to more easily compare the impact strengths of the diffusion bonds with those of the parent metal controls, normalized impact strengths are plotted against bonding temperature (Fig. 6). The normalized impact strength is defined as the ratio of the mean impact strength of diffusion-bonded specimens to that of control specimens for a particular set of bonding parameters. It is evident that although the measured impact strengths of the diffusion bonds reach a maximum at 900° C and then decrease with increased temperature, the normalized impact strengths increase with bonding temperature.

The parent metal control specimens in general

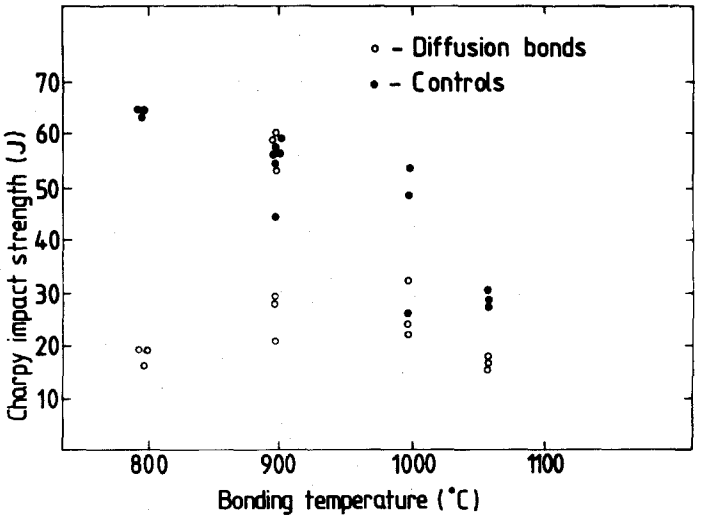

Figure 5 Variation of impact strength with bonding temperature (diffusion bonds in En8-A with a bonding time of 20 min at a pressure of 20 MN m^{-2}, impact test temperature at 80° C).

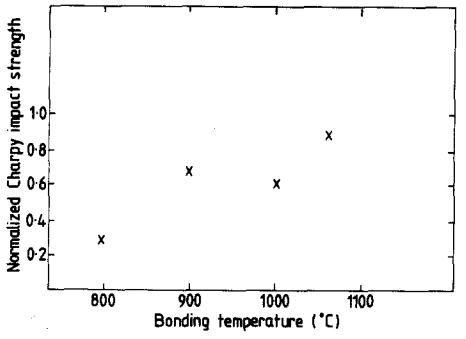

Figure 6 Variation of normalized impact strength with bonding temperature for results shown in Fig. 5.

failed by ductile fracture to give a fracture surface containing large shear lips. As the bonding temperature increased, the extent of cleavage failure increased due to an increase in grain size and consequent increase in the ductile/brittle transition temperature. In comparison, the diffusion-bonded specimens failed by flat fracture at or adjacent to the bond line. Failure at the bond line was by ductile fracture of bonded regions whilst failure in metal adjacent to the bond line was by cleavage. As the bonding temperature increased from 800° C to 1060° C, the amount of cleavage failure increased from 0% to 80%.

3.3.2. Variation of impact strength with post-bond heat treatments

Diffusion bonds fabricated in En8-A at 900° C and given the post-bond heat treatments previously described (Section 2.2), were tested both at room temperature and at + 80° C; the results are shown in Figs. 7 and 8, respectively. For the specimens tested at room temperature, although there is a large spread in the values of the impact strengths obtained, the impact strengths appear to be of similar magnitudes. The scatter in the results is due to the testing temperature being in the ductile/brittle transition range for En8. In contrast, the impact strengths of bonded specimens tested at + 80° C remain in all cases less than those for the control specimens; the post-bond thermal treatments appear to have little effect. This is shown more clearly by plotting normalized impact strength against post-bond heat treatment (Fig. 9).

For the specimens tested at + 80° C, the control specimens failed by ductile fracture whilst the bonded specimens gave flat fracture surfaces. The flat fracture surfaces consisted mainly of ductile failure at the bond line together with some cleavage failure in metal adjacent to the bond line. For the specimens tested at room temperature, the control specimens failed by mixed ductile and cleavage fracture whilst the bonded specimens failed by bond line ductile fracture with cleavage in metal adjacent to the bond line.

3.4. SEM examination of fracture surfaces

3.4.1. Tensile tests

The diffusion-bonded specimens and the control specimens all failed giving typical ductile cup and cone-type fractures. The diffusion-bonded specimens fractured in metal away from the bond interface.

3.4.2. Impact tests

The parent metal control specimens which failed in a ductile manner gave fracture surfaces containing large shear lips at the edges while in the

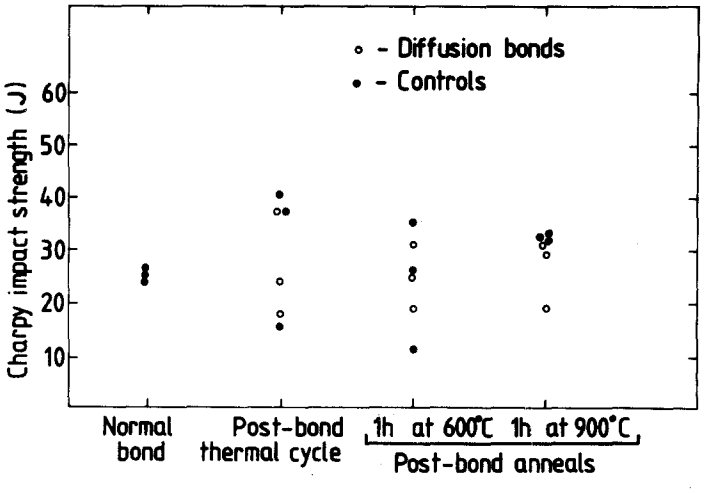

Figure 7 Variation of impact strength with post-bond heat treatments (diffusion bonds in En8-A bonded at 900° C for 20 min at a pressure of 20 MN m^{-2}, impact tested at room temperature).

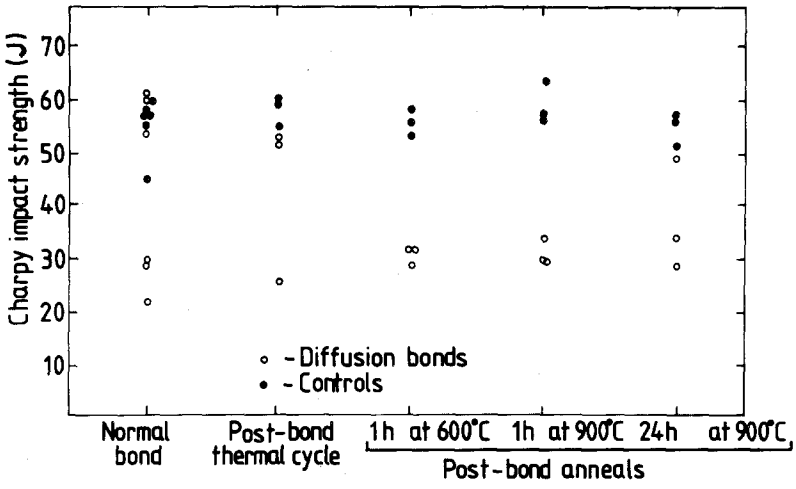

Figure 8 Variation of impact strength with post-bond heat treatments (bonding conditions as Fig. 7, impact test temperature of 80° C).

centres both ductile failure (by void formation) and some secondary cracking was visible.

The flat fracture surfaces shown in Fig. 10 are typical of those observed after impact testing diffusion-bonded specimens. It can be seen that the regions of ductile failure which characterize bonded areas are separated by grooves. These grooves are parallel to each other and to the direction of the original grinding marks and thus represent unbonded regions. Thermal etching of grain boundaries was seen in the regions of unbonded area confirming that non-bonded regions act as free surfaces during the bonding process. Since the unbonded regions are present as grooves or cylinders through the structure, the bond line voids observed in metallographic mounts (Section 3.1) are sections through these cylinders which lie parallel to the direction of the original grinding marks. Cleavage failure occurred in parent metal adjacent to the bond line when the crack propagated away from the bond line.

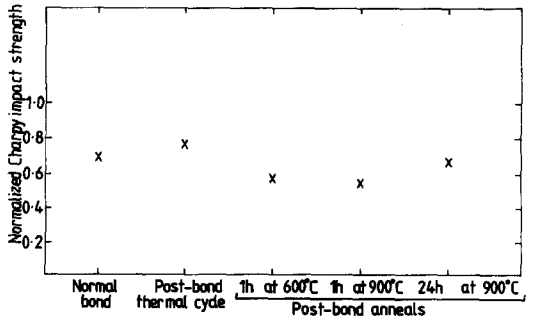

Figure 9 Variation of normalized impact strength with post-bond heat treatments for results shown in Fig. 8.

4. Discussion

4.1. Microstructure at the bond line

From scanning electron microscope studies of the fracture surface of diffusion-bonded specimens, it is apparent that the voids visible in metallographic sections are cylinders lying parallel to the direction of the original grinding marks. There is a large decrease in void density with a post-bond annealing heat treatment even though there is only a small decrease in void size. The large scatter in the void size measurements is attributed to:

(1) scatter in the size of the voids;

(2) errors due to the small number of voids measured;

(3) smallness of the voids being measured;

(4) selectivity as regards to which voids were photographed.

Voids were seen to occur both in regions of ferrite and pearlite for all bonds fabricated at or above 900° C. This suggests that austenite grain growth across the bond line occurred to a certain extent at these bonding temperatures.

Optical microscopy of the diffusion-bonded specimens showed that, in many cases, the bond line was delineated by an essentially planar layer of ferrite; this has also been reported by Elliott [3], Signes [4] and Taylor and Pollard [9]. It is assumed that during bonding the two contacting surfaces form a planar grain boundary in the austenite phase. On cooling, ferrite nucleates at austenite grain boundaries and thus the bond line becomes delineated by a planar layer of ferrite. Although the prior austenite grain size was observed to increase as the bonding temperature increased, grain growth of the austenite did not occur across the boundary, probably because it was pinned

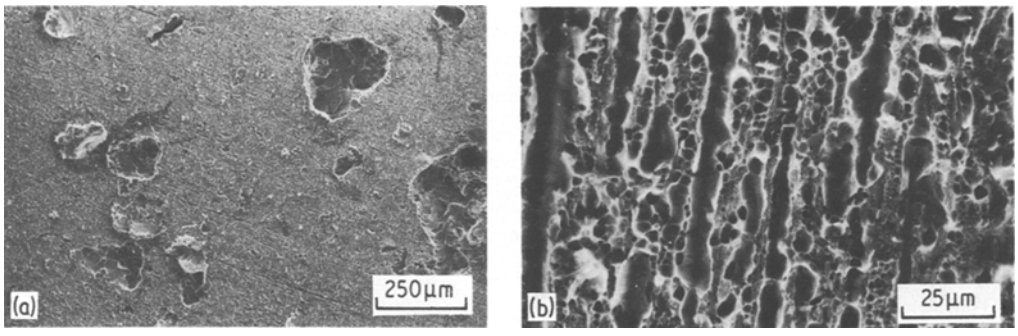

Figure 10 SEM micrographs of fracture surfaces of En8 bonded for 20 min and 20 MN m^{-2} at temperatures of: (a) 900° C; (b) 800° C.

by contaminant, voids and/or inclusions. At a bonding temperature of 1060° C, massive grain growth occurred across the boundary so that subsequently the bond line was no longer delineated by a planar layer of ferrite.

It was shown that the planar layer of ferrite was not present at the bond line for specimens given a post-bond heat treatment which involved re-austenitizing at 900° C. On re-heating to or above 900° C, the austenite grains can nucleate at the bond line and then grow across it so that the original planar austenite grain boundary is removed. Consequently, on cooling, ferrite will no longer nucleate at the bond line to form a planar layer.

4.2. Tensile tests

All the specimens tested achieved tensile properties equivalent to those of the control specimens. Although a planar layer of ferrite was present at the joint interface (except for specimens bonded at 1060° C), it did not have a detrimental effect on the tensile properties. This is in contradiction to Elliott [3] who claimed that the removal of the layer of ferrite was necessary to promote failure away from the bond line. All the specimens tested failed by ductile fracture away from the bond line and gave satisfactory reduction in area values at fracture even though complete bonding had not taken place at the interface. From other work performed on En8 by Thornton [6], it has been shown that, under the bonding conditions used, the interface contains at least 80% bonded area in all cases. This is in agreement with the results of Elliott *et al.* [2] who showed that parent metal tensile properties are achieved even when the interface contains only 80% bonded area.

4.3. Impact tests

This investigation has provided data on the impact strengths of diffusion bonds in En8. It has also been possible by the use of selected post-bond heat treatments to start to investigate the hypotheses (Section 1) proposed to account for the observed low impact strengths.

For all the impact specimens tested, the impact strengths of the diffusion-bonded specimens were less than those of the parent metal control specimens. Also, for all bonding temperatures used, the impact energies were similar to those obtained by Elliott [3] (even though the bonding conditions were not exactly equivalent to those used by Elliott) although the values of impact strength obtained for a particular bonding condition show less scatter than do those reported by Elliott. This may be attributed to the fact that Elliott did not use a constant notch position relative to the direction of the original grinding marks.

The impact strengths of bonds fabricated at different bonding temperatures in En8-A show a peak in impact strength at 900° C. Above this temperature, the impact strength decreased due to an increase in grain size and consequent increase in the amount of cleavage failure. The decrease in impact strength of the parent metal controls with increasing bonding temperature is also attributed to the increase in grain size. Diffusion-bonded specimens failed by ductile fracture at the original bond line and by cleavage failure in metal adjacent to the bond line. Failure at the bond line was observed even on specimens in which some grain growth and pearlite colonies were observed to cross the boundary.

The post-bond heat treatments were found to have no effect on the impact strength of diffusion bonds fabricated in En8-A at 900° C and this

result again is in contrast to that of Elliott [3] who found that a post-bond annealing heat treatment raised the impact strengths of the diffusion bonds to that of the parent metal. The elimination of the planar layer of ferrite at the bond line had no effect on the impact strength of the diffusion-bonded specimens, i.e. the planar layer of ferrite hypothesis (Section 1) is not substantiated. Likewise, the reduction in void density at the bond line due to post-bond annealing had no effect on the impact strength of the diffusion-bonded specimens. Thus in order to achieve good impact properties, it may be necessary to remove the bond line voids completely and not simply to reduce the density of the voids.

In view of the above results, it is thought that the weakness of the bond line may be attributed to an increased inclusion density at the bond line (compared to that in the parent metal), to the segregation of the embrittling elements from the bulk of the specimen to the bond line, or to the presence of microvoids remaining at the bond line.

5. Conclusions

The work described has shown that it is not possible for diffusion bonds fabricated in En8, under the bonding conditions used, to achieve parent metal impact strengths although it is possible for them to achieve parent metal tensile strengths. Consequently, impact testing has been confirmed to be a more severe test of the quality of diffusion bonds than tensile testing. The other main conclusions are:

(1) the removal of the planar layer of ferrite at the bond line does not result in an improvement in impact strength;

(2) a decrease in the density of bond line voids, i.e. an increase in fractional bonded area, does not result in an improvement in impact strength;

(3) flat fracture occurs at the bond line when the impact test is carried out above the ductile/brittle transition temperature and this is believed to be due to the presence of a high inclusion or microvoid density and/or segregant at the bond line;

(4) cleavage failure occurs away from the bond line when the impact test is carried out below the ductile/brittle transition temperature.

Current work is in progress examining the remaining hypotheses summarized in the Introduction.

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