Chapter 19
Investigation on the Mechanical and Microstructural Characteristics of Diffusional Bonded Gray Cast Iron and Low Carbon Steel

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Abstract An investigation has been made of the diffusional bonded couples of gray cast iron and low carbon steel that has been subjected to heat treatment. The objective is to establish the post bond heat treatment's parameters influence on the mechanical and structural properties of the diffusional bonded couples. The tensile strength of the diffusion bonded joints was found to be increased with increased heat treatment's temperature and time. The microstructural examination has also shown that at higher heat treatment temperature and time also had resulted in microvoids and interface lines to be disappeared, and the bond/weld at the interfaces of the diffusional welded couples seemed to be more complete. Correspondently, these resulted in the increased of the tensile strength. The microhardness value at the interface lines of the joints was found to be increased, while the charpy impact strength value was on the opposite way with the increased heat treatment temperature. The microstructural analysis has also shown that much thicker diffusion layers of spherodization zone and carbon rich zone were formed at higher temperature at the interfaces of the

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joints. These are also correlated with the microhardness and the charpy impact values obtained at higher temperature. These results were in consistent with the principle and theory of diffusion bonding whereby at higher temperatures, more activation energy is available for atoms inter-diffusion to take place, while with longer time, it allows higher volume of diffusion of atoms, and hence it changes the behavior of the joints. Thus, heat treatment's temperature and time were found to have a strong influence on the mechanical and microstructural characteristics of the diffusion bonded gray cast iron and low carbon steel.

Keywords Diffusion bonding · Heat treatment · Mechanical · Microstructure · Diffusion layer

19.1 Introduction

Diffusion welding is a joining process between materials wherein the principal mechanism for joint formation is a solid-state diffusion. Coalescence of the faying surface is accomplished through the application of pressure at elevated temperature. There is no melting and only limited macroscopic deformation, or relative motion of the parts occurs during welding [1]. Diffusion welding is an attractive joining technique for the manufacture of precision apparatus and complex structures [2]. Diffusion welding helps to join difficult-to-weld materials or whenever standard fusion welding methods are unable to be used [3].

The success or failure of the diffusion welding process depends on three variables that require constant watch and careful adjustment. These variables are the welding temperature, the welding pressure (or pressing load), and the holding time (duration of pressure) [1].

A conventional diffusion welding process is usually carried out in a vacuum environment where the mating surfaces are not only protected against further contamination, such as oxidation, but remain clean due to the dissociation, sublimation or dissolution of the oxides present that diffuse into the bulk of the materials [1]. A conventional diffusion welding experiment carried out is using the typical equipment outfitted with the pressurized, heating and vacuum systems.

Various alternative methods were tested in previous studies to perform diffusion welding experiments to seek for an alternative to the conventional method of using the standard machine/equipment known as the hot press used in the preliminary study that had broken down and been rendered unusable [4].

Finally, the equipment and method as described in the previous study are selected for this research. Despite using this method, the results of the tensile test revealed that the joint was weak, however, this method is still successful in producing diffusion welded couples. In the following experiments, the diffusion couples produced through this method are subsequently be placed inside the furnace again without clamping on the fixture for a post bond heat treatment (PBHT). It is expected that with further heat treatment, it would remove voids at the interfaces of the joints and complete the bond/weld by allowing further diffusion process to take place. The objective is to study the effect of heat treatment temperature and time on the diffusion process and the characteristic of the joint. Tensile testing, Charpy impact testing, microhardness and metallographic examination are conducted.

19.2 Methodology

19.2.1 Materials

Cast iron, containing lamellar graphite of grade ASTM A 48 class 35 and equivalent to the ISO 250 and EN-GJL-250, is used with chemical composition and mechanical properties as shown in Tables 19.1 and 19.2, respectively.

Low carbon steel of grade BS 449 grade 250, equivalent to that of ASTM 36 and JIS G 3101 SS 400, is used with chemical composition and mechanical properties as shown in Tables 19.3 and 19.4, respectively.

Table 19.1 Composition of grey cast iron

	Chemical composition (wt. %)				
	C	Si	Mn	P	S
Grey cast Iron (ASTM A48C 35)	3.43	2.21	0.62	0.073	0.069

Table 19.2 Mechanical properties of cast iron

	Tensile strength (MPa)	Elongation (%)	Hardness (HB)
ASTM A48 C 35	250	–	190

Table 19.3 Composition of low carbon steel

	Chemical composition (wt %)				
	C	Si	Mn	P	S
BS 449 grade 250	0.19	0.10	0.46	0.011	0.031

Table 19.4 Mechanical properties of low carbon steel

	Tensile strength (MPa)	Elongation (%)	Hardness (HB)
BS 449 grade 250	408	35	195

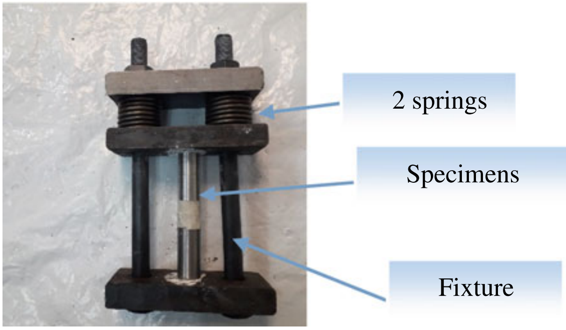


Fig. 19.1 Specimens clamped in fixture

Fig. 19.2 Hydraulic Press with specimens clamped in fixtures

19.2.2 Diffusion Bonding Equipment

Non-standard equipment/unconventional methods were developed to conduct diffusion bonding experiments [4]. In this method, a fixture was developed to clamp the specimen as shown in Fig. 19.1. A press machine was fabricated as in Fig. 19.2 to transfer pressure or load to the specimens. In this way, the specimens were pre-pressed with load before placing them into the heating furnace. Atmospheric furnace, as shown in Fig. 19.3, was used. The heating system is of radiation in nature that consists of a coil resistance furnace as the heat source, with appropriate switches and instrumentations needed to measure and monitor the desired treatment temperature and time.

19.2.3 Specimen Preparation

The specimen materials were cut out on a lathe machine into cylindrical shapes of size ø15 × 50 mm. The specimen ends were joined and polished on 240, 600 and

Fig. 19.3 Atmospheric furnace

Table 19.5 Diffusion bonding parameters and conditions

Welding pressure (MPa)	Bonding temp. (°C)	Bonding time (Min)
14	900	60

800 grits abrasive paper and cleaned using alcohol to remove any loose grit, dirt and grease or other contaminants.

19.2.4 Diffusion Bonding Parameters and Conditions

Welding parameters and welding conditions were selected based on best of past experiments and related literature reviews.

The welding process was conducted under atmospheric condition. The welding pressure selected was higher than the one usually carried out under vacuum in the preliminary study. At a higher pressure, it is evident that welding pressure plays an important role in the blocking of oxidation at the weld interface under atmospheric pressure [5].

Experiments to produce diffusion couples were carried out using the welding conditions as shown in Table 19.5.

19.2.5 Diffusion Bonding Procedure

The specimens were mounted, firstly, on fixtures as in Fig. 19.2. Loads or pressures were then transferred to mating surfaces through the fixtures using the press machine as shown in Fig. 19.2. The specimens, together with the fixtures, were placed in the furnace as shown in Fig. 19.3. The heating temperature, duration and heating rate were set through the control panel.

Upon heating and reaching the set temperature, the specimens were subsequently held for the set duration. At the end of the holding time, the heating was stopped automatically to allow the temperature to reduce by itself. The specimens are cooled to less than 1000 °C before removing from the furnace to avoid further oxidation.

19.2.6 Post Bond Heat Treatment

Diffusion couples produced using the above method were subsequently be placed into the furnace again without clamping in the fixture for a post bond heat treatment (PBHT). It is expected that with further heat treatment, voids would be removed at the interfaces of the joints to complete the bond/weld by allowing further diffusion process to take place.

19.2.7 PBHT for Tensile Testing

(a) Effect of PBHT temperature: Experiments were carried out using the PBHT conditions as shown in Table 19.6.
(b) Effect of PBHT time: Experiments were carried out using the PBHT conditions as shown in Table 19.7.

Table 19.6 PBHT parameters and conditions

Temperature (°C)	Time (h)
750	2
850	2
950	2
1000	2

Table 19.7 PBHT parameters and conditions

Temperature (°C)	Time (h)
1000	1
1000	2
1000	4
1000	8

Table 19.8 PBHT parameters and conditions

Temperature (°C)	Time (h)
800	2
900	2
1000	2

Table 19.9 PBHT parameters and conditions

Temperature (°C)	Time (h)
1000	1
1000	2
1000	4

Table 19.10 PBHT parameters and conditions

Temperature (°C)	Time (h)
800	2,4,8
900	2,4,8
1000	2,4,8

19.2.8 PBHT for Charpy Impact Testing

(a) Effect of PBHT temperature: Experiments were carried out using the PBHT conditions as shown in Table 19.8.
(b) Effect of PBHT time: Experiments were carried out using the PBHT conditions as shown in Table 19.9.

19.2.9 PBHT for Metallographic Examination and Microhardness Testing

Experiments were carried out using the PBHT conditions as shown in Table 19.10.

19.2.10 Tensile Testing

Tensile test was conducted on the diffusion couples after PBHT based on the standard DIN 50125, type F test piece, whereby the specimens were in the original form, round bar and un-machined conditions. In this test, the gage length taken was 25 mm, while the rate of extension/crosshead speed is 1 mm per min. An instron universal testing machine was used.

19.2.10.1 Charpy Impact Testing

The Charpy impact test was conducted on the diffusion couples after PBHT based on the standard ASTM E23. ASTM E23-16b as a guideline including the specification of specimen, procedure of testing, safety precaution and data collection. The test specimen is thermally conditioned and positioned on the specimen supports against the anvils; the pendulum is released without vibration and the specimen is impacted by the striker. Information is obtained from the machine and from the broken specimen.

19.2.10.2 Microhardness Testing

The metallographic specimens were also used for hardness testing. In this test, the microhardness tester of the Vickers hardness testing machine was employed with loads of 25 g (gf). The hardness was measured across the bonding interface.

19.2.10.3 Metallographic Examination

After completing the PWHT, the specimens were then prepared for a metallographic examination. They were mechanically polished on succession of 120, 240, 360 and 600, 800 and 1200 grits emery papers before final polishing on metallographic cloths moistened with a diluted solution of diamond particles of 3 and 1 μm sizes successively. The specimens were then etched initial solution (100 ml methanol + 4 ml nitric acid).

Photographs of the prepared metallographic specimens, in the vicinity of diffusion zones, along the bonding interface were then taken by optical microscope.

19.3 Results and Discussion

Diffusion couples were produced arising from the diffusion welding experiments. Figure 19.4 shows one of the diffusion couples still attached to the fixture after removal from the furnace.

Figure 19.5 shows one of the diffusion couples after removal from the fixture. Visual examination and manual testing on the diffusion couples revealed that the joints look good and strong.

(a) Effect of PBHT Temperature

The effect of PBHT temperature on tensile strength of the diffusional bonded joints is shown in Fig. 19.6. The tensile strengths of the joints increased directly with PBHT temperature despite the value being low but slightly increasing.

A typical stress–strain graph of the effect of PBHT's temperature is as shown in Fig. 19.7. Highest tensile strength obtained was 14.71 MPa, that being considered

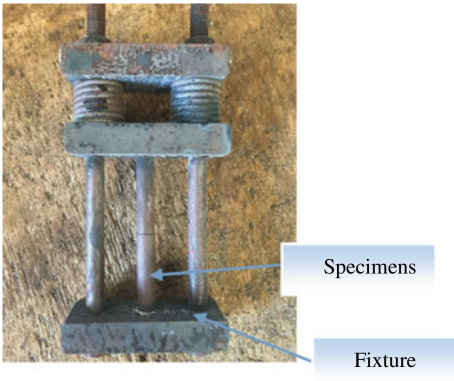


Fig. 19.4 Diffusion couple after removal from furnace

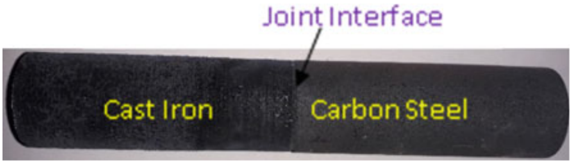


Fig. 19.5 One of successful joint specimens of grey cast iron and low carbon steel (Tw = 900 °C, tw = 60 min, Pw = 13 MPa)

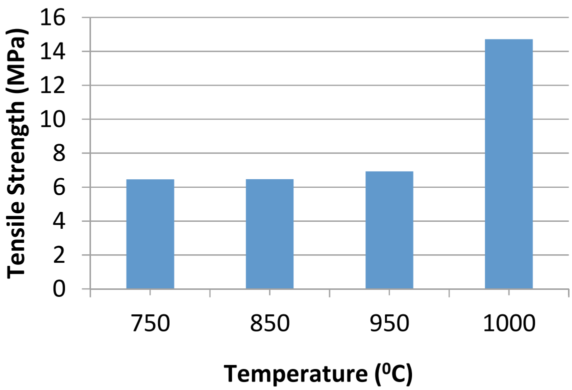


Fig. 19.6 Tensile strength of joints as a function of PBHT temperature

as low, while the tensile strength for gray cast iron, being the lowest material, is 250 MPa. The elongation rate was also very minimal.

A typical fractured surface after a tensile test as function of PBHT's temperature is shown in Fig. 19.8. Bonding or diffusion only occurred at the side of joint interfaces, while no diffusion took place at the center. Effect of PBHT time: The effect of PBHT time on tensile strength of the diffusional bonded joints is shown in Fig. 19.9. The tensile strengths of the joints increased directly with PBHT time despite the value being low but slightly increasing.

A typical stress–strain graph of the effect of PBHT's time is shown in Fig. 19.10. The highest tensile strength obtained was 11.05 MPa and can be considered as low,

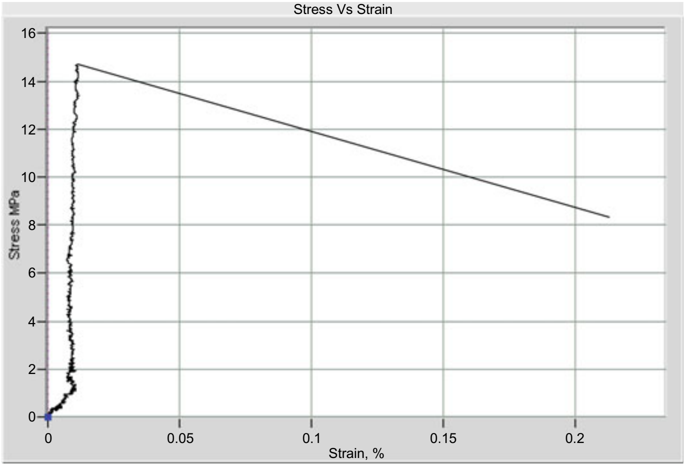


Fig. 19.7 Stress versus Strain graph of effect of temperature

Fig. 19.8 Typical fractures on surfaces of specimen after tensile test

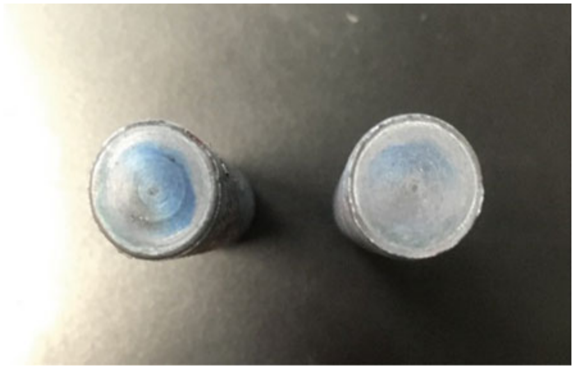
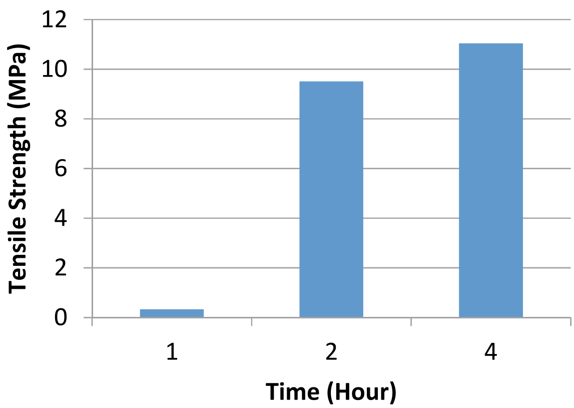


Fig. 19.9 Tensile strength of joints as a function of PBHT time



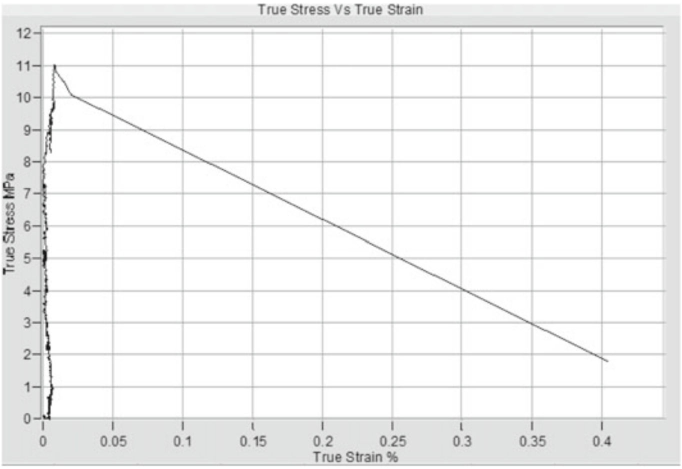


Fig. 19.10 Stress versus Strain graph of effect of time

as tensile strength for the lowest material, gray cast iron, is 250 MPa. The elongation rate was also very minimal.

A typical fractured surface after a tensile test as function of PBHT's time is shown in Fig. 19.11. Bonding or diffusion only occurred at the side of joint interfaces while no diffusion took place at the center.

Effect of PBHT temperature: The effect of PBHT temperature on the Charpy impact value of the diffusional bonded joints is shown in Fig. 19.12. The Charpy impact value of the joints is decreased with PBHT's temperatures and the value is very low as compared to the base metal.

Effect of PBHT time: The effect of PBHT time on the Charpy impact value of the diffusional bonded joints is shown in Fig. 19.13. The Charpy impact value of the joints is also very low and is of reduced value with PBHT times.

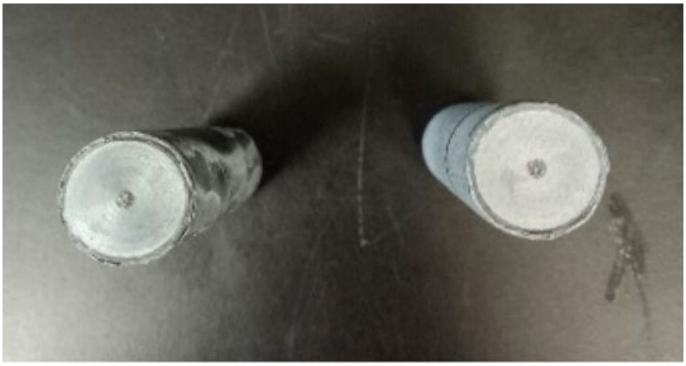


Fig. 19.11 Typical fractures on surfaces of specimen after tensile test

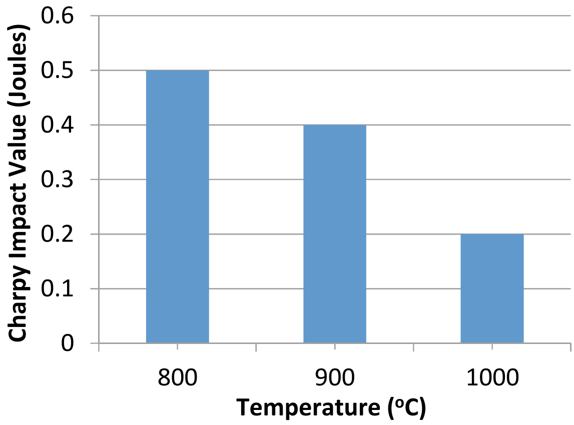


Fig. 19.12 Charpy impact value of joints as a function of PBHT temperature

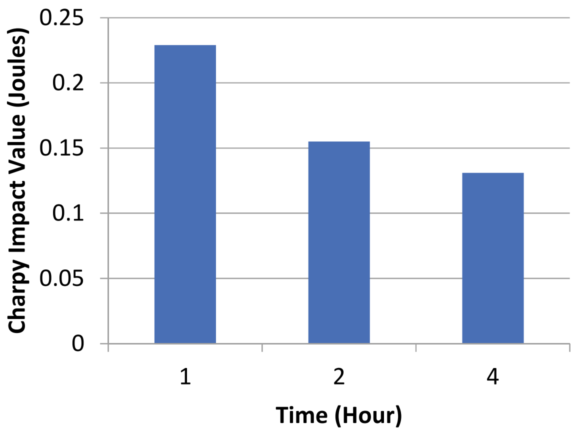


Fig. 19.13 Charpy impact value of joints as a function of PBHT time

The effect of PBHT temperature and time on the microhardness of the diffusional bonded diffusion layers at the interface lines of the joints is as shown in Fig. 19.14. The microhardness of the joints significantly increased directly with PBHT temperature but remained almost constant with the time.

A typical micro-Vickers hardness gradient across the bonding interface of gray cast iron and low-carbon steel diffusion couples after 2, 4 and 8 h at 1000 °C is shown in Fig. 19.15. The hardness of the diffusion layer produced is much higher than the hardness of the respective base metals at all treatment times. The maximum hardness was observed close to the interface on both sides of cast iron and low carbon steel. The maximum hardness was mainly at the interface line as shown in Fig. 19.14.

Figure 19.16 shows the typical optical micrograph of the joint interfaces; of (a) 800 °C (b) 900 °C and (c) 1000 °C for 4 h PBHT before etching. Microvoids and interface lines are very much visible at the interfaces of the specimens treated at temperature of 800 °C, becoming less visible treated at 900 °C and almost disappear

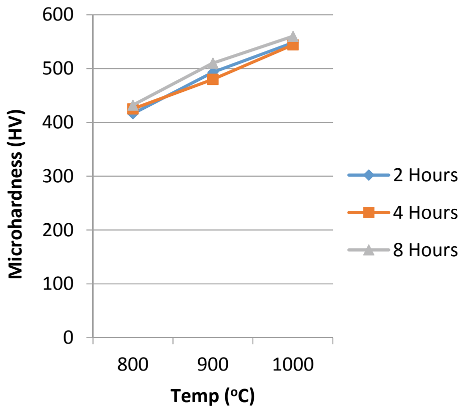


Fig. 19.14 Microhardness of diffusion layer as a function of PBHT temperature at the interface line

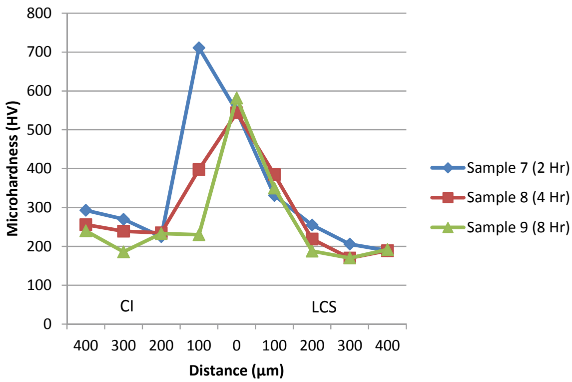


Fig. 19.15 Microhardness profile across the interface of diffusion couples after PBHT (1000 °C) [6]. Reprinted with the permission of IOP Publishing

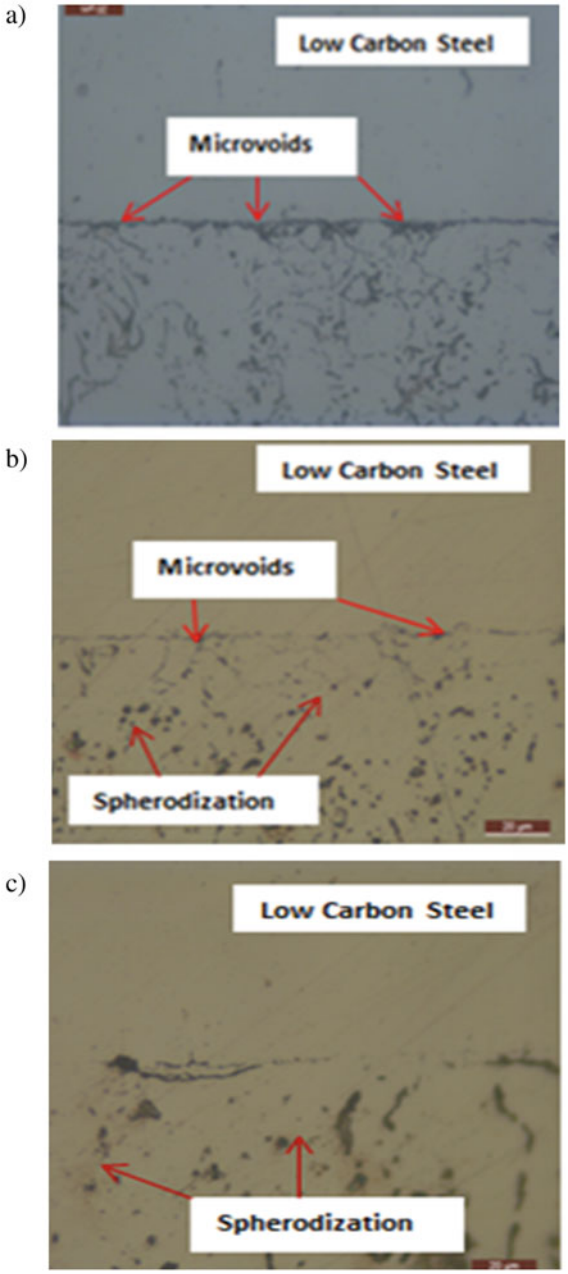
and the bond/weld also seems to be more complete treated at 1000 °C. Spherodization or dissolution of graphite flakes is observed on the cast iron sides close to the interface of 900 and 1000 °C treated.

Figure 19.17 shows the typical optical micrograph of the joint interfaces; of (a) 800 °C (b) 900 °C and (c) 1000 °C for 4 h PBHT after etching. From Fig. 17a, a diffusion layer of carbon rich zone has formed but spherodization or dissolution of graphite flakes was not observed on the cast iron sides close to the interface. From Fig. 17b, c spherodization or dissolution of graphite flakes was observed on the cast iron sides close to the interface of specimens. As a result of the above, diffusion layers of spherodization zone and carbon rich zone have formed near the interface of cast iron and low-carbon steel side, respectively.

The effect of PBHT temperature and time on the thickness of diffusion layers is shown in Fig. 19.18. A steady increase in thickness of diffusion layers is observed as diffusion temperature and time consecutively increase.

The diffusion coupled of gray cast iron and low carbon steel was successfully produced by the alternative diffusion welding method and equipment described in

Fig. 19.16 Optical Mocrosopic of the joint interface at **a** 800 °C, **b** 900 °C and **c** 1000 °C for 4 h PBHT before etching [6]. Reprinted with the permission of IOP Publishing



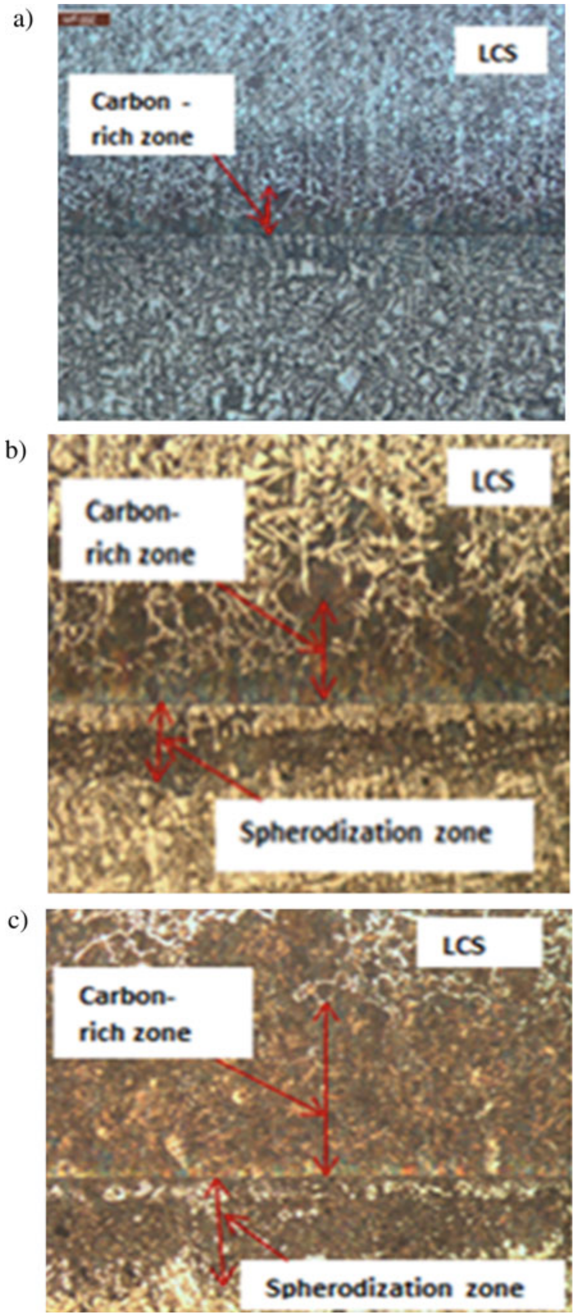


Fig. 19.17 Optical microscopic of the joint's interface at **a** 800 °C, **b** 900 °C and **c** 1000 °C for 4 h PBHT after etching [6]. Reprinted with the permission of IOP Publishing

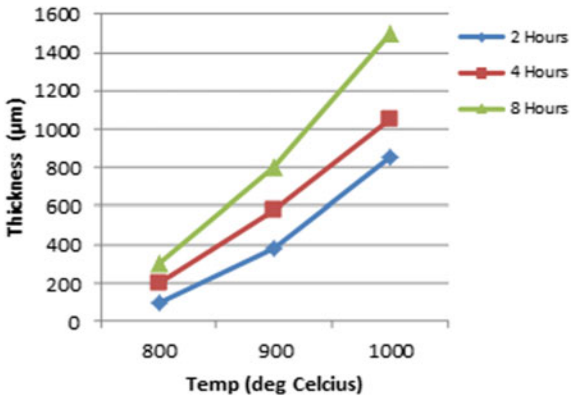


Fig. 19.18 Thickness of diffusion layer [6]. Reprinted with the permission of IOP Publishing

the procedure. Later, post weld heat treatment (PBHT) was conducted to continue the diffusion process to achieve the complete bond. The results obtained from the tensile testing as shown in Figs. 19.6 and 19.9 can be deduced that at higher temperature and longer duration of PBHT had resulted in an increase in tensile strength of the joints. This increase in the strength is correspondent with the reducing and elimination of voids and interface lines observed in the optical microscopic graphs as shown in Fig. 16a–c that complete the bond. This phenomenon of elimination of the interfacial defects (voids) and the original interface at higher diffusion temperatures were also observed in the diffusion bonding of cast iron to medium carbon steel [7].

A steady increase in thickness of diffusion layers is also observed as diffusion temperature and time consecutively increase as shown in Fig. 19.18. All these phenomena is consistent with the principle and theory of diffusion bonding whereby at higher temperature, more activation energy is available for atoms inter-diffusion to take place, while with more time duration will allow more volume of diffusion of atoms, hence improving the bond and the strength of the joints [8].

Meanwhile the hardness of the diffusion layers increased with an increase in temperatures as shown in Fig. 19.14. This is correspondence to the area of diffusion layers of spherodization zone and carbon rich zone formed at higher temperatures as shown at Fig. 17a–c. The hardness across the diffusion layers has it ultimate values at the interface lines and reduced toward the base metals. The above high hardness value can be related to the presence of high amount carbides. According to the researchers [9], increasing hardness value is due to the higher the temperature and the longer times available for carbon to diffuse. This is also correspondently with the decreased of charpy impact values with the increasing times as in Fig. 19.13, which means also increase in brittleness as hardness increases.

The natures of the elements or compositions, the compounds formed that contributed to the very high hardness and brittleness of the diffusion layers especially along the interface lines will be examined in future analysis using scanning electron microscopic (SEM) and the X-Ray diffractometer (XRD). This high hardness values with increased temperature and the formation of carbide at the interface is correspondent with the very low and decreased of charpy impact values at increased temperatures as obtained in Fig. 19.12.

The values, of the tensile and charpy impact strengths as obtained in the results and as discussed above, were found to be low as compared to the base metals. Arising from the fractured surface of the tensile and charpy impact tests specimens it was observed that the bonding or diffusion only occurred at the side of joint interfaces, while there was no diffusion at the center. This effect could be due to the wide gaps or voids still present at the center of the joint interfaces after diffusion bonding prior to the PBHT. The reasons for the incomplete bond of the diffusion bonded couples produced is consistent with the fundamental reason of diffusion welding experiments that were conducted previously but had failed to produce quality joints, namely, due to the welding pressure not being adequately applied, arising from the loss of pressure caused by thermal expansion of the specimens and the fixtures [10]. As weld or bond had still occurred, though minimal, inter-diffusion of atoms across the interfaces was also found to be very minimum. If the pressure is insufficient, some of the voids will be left unfilled, and the strength of the joint will be impaired, to the extent that the bond or joint may not form [8].

Voids at the interface of the joints were not fully eliminated and resulted in complete bond could also be attributed to poor surface preparation [2].

Nevertheless, the subsequent PBHT subjected on the diffusion couples was not fully capable to facilitate further inter-diffusion of atoms across the interfaces to produce complete bond and strong joints.

19.4 Conclusion

The diffusion couples of gray cast iron and low carbon steel were successfully produced by the alternative diffusion welding method and equipment described in the procedure. This allows for the continuance of this research to be done on the diffusion welding and subsequent investigation on the mechanical and metallographic characteristics by conducting PBHT on the diffusion couples. From the tensile, Charpy impact and microhardness test results, a direct correlation between the PBHT's temperature and time and the mechanical strengths was observed. Comparably, the metallographic examinations result also showed a direct correlation with PBHT's parameter. It could be concluded that at higher temperature and longer duration of PBHT would have a strong influence on both the mechanical and the metallographics characteristics of the joints. This is consistent with the principle and theory of diffusion welding whereby at higher temperature, more activation energy is available for atomic inter-diffusion to take place, while with more time duration will allow more volume of diffusion of atoms, hence improving the bond and the strength of the joints.

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References

1. Kazakov NF (1985) An outline of diffusion bonding in vacuum. In: Diffusion bonding of materials. Pergamon Press, Oxford
2. Airu W, Osamu O, Kenji U (2006) Effect of surface asperity on diffusion bonding. Mater Trans 47:179–184
3. Kolarik L et al (2015) Influence of diffusion welding time on homogenous steel joints. Procedia Eng 100:1678–1685
4. Ayob F et al (2020) Unconventional approach in the study of diffusion welding of marine grades grey cast iron to low carbon steel. Mater Today Proc 29:179–174
5. Momono T et al (1990) Diffusion bonding of cast iron to steel under atmospheric pressure. Cast Sci 26:72
6. Ayob F et al (2018) Effect of post heat treatment on the microstructure and microhardness of diffusion coupled gray cast iron and low carbon steel. IOP Conf Ser: Mater Sci Eng. https://doi.org/10.1088/1757-899X/328/1/012001
7. Calvo FA et al (1989) Diffusion bonding of grey cast iron to ARMCO iron and a carbon steel. Mater Sci 24:4152–4159
8. Kazakov NF (1985) Bonding of cast iron and cast iron to steel. In: Diffusion bonding of materials. Pergamon Press, Oxford
9. Abdullah AA, Rawhdan RR (2014) Microstructure characteristics in the interface zone of gray cast iron solid state bonds. Eng Tech J 32:15–22
10. Agilent Technologies (2007) Laser and optics: user's manual. http://www.uzimex.cz/soubory/20080403_laser_optika_cast_1.pdf. Accessed 20 May 2021