# Examination of Micro-structural, Mechanical Properties and Investigation of Optimum Conditions of Diffusion Bonding between *Grey Cast Iron* and *Low Carbon Steel*

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Keywords: Diffusion Bonding, Microstructure, Tensile Test, Micro-hardness Test, Heat Treatment.

Abstract: An experimental research is being conducted on the joining of dissimilar materials of grey cast iron to low carbon steel utilizing diffusion bonding method. Diffusion bonding process operates on the principle of solid-state diffusion, wherein the atoms of two solid, metallic surfaces intersperse themselves over time, moving an atomic mass form or diffusion through the lattice of a crystalline solid. Upon producing of the diffusion couples through diffusion bonding, the bonds are subjected to SEM (Scanning Electron Microscopy), EPMA (Electron Probe Micro Analyzer), XRD (X-Ray Diffraction), EDS/EDX (Energy Dispersive X-Ray Spectroscopy), WDX's (Wavelength-dispersive X-ray spectroscopy) micro-structural analysis and mechanical properties examination. Subsequent investigation is to be carried out to establish the diffusion mechanism, inter-diffusion coefficients and activation energy of the diffusion system. To study the optimum conditions that produce excellent and ultimate bond, various bonding parameters and variables are taken into consideration. This paper describes the research progress undertaken to date, explaining the materials involved, equipment, method and preliminary as well as current results on microstructure analysis, tensile test and micro-hardness test.



## 1 INTRODUCTION

In this experimental research, materials of cast iron and carbon steel are chosen as they are abundantly used in the various industries (B. Kurt *et al.*, 2007), yet they are difficult to join by the conventional fusion welding due to chemical, mechanical and structural heterogeneities associated with fusion welding N. Fujii *et al.*, 2006).

Diffusion bonding is being viably predicted for application in the joining of cast iron to low carbon steel based on the principle of solid-state diffusion, wherein the atoms of two solid, metallic surfaces intersperse themselves over time. Diffusion process is actually the atomic movement of mass form or diffusion through the lattice of a crystalline solid.

Steady state diffusion is determined by the amount of diffusion flux that passes through the cross-sectional area of the mating surfaces of the couple. Fick's first law of diffusion states:

$$J = -D\,(dC/dx) \quad (1)$$

where J is the diffusion flux, D is a diffusion coefficient, and dC/dx is the concentration gradient through the materials in question. The negative sign is a product of the gradient. Another form of Fick's law states:

$$J = M/(At) \quad (2)$$

where M is defined as either the mass or amount of atoms being diffused, A is the cross sectional area, and t is the time required. Equating the two equations and rearranging, we achieve the following result:

$$t = -(1/D)(M/A)(dC/dx)^{-1} \quad (3)$$

As mass and area are constant for a given joint, time required is largely dependent on the concentration gradient, which changes by only incremental amounts through the joint, and the diffusion coefficient. The diffusion coefficient is determined by the equation:

$$D = D_0 e^{-Q_d/RT} \quad (4)$$

where $Q_d$ is the activation energy for diffusion, R is the universal gas constant, T is the temperature



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experienced during the process in Kelvin, and $D_0$ is a temperature-independent preexponential that depends on the materials being joined. For a given joint, the only term in this equation within control is temperature (Callister *et al.*, 2014).

The activation energy for atomic diffusion at the surface, interface and grain boundaries is relatively low compared to the bulk diffusion (diffusion through crystal lattice) due to a looser bond of the atoms and higher oscillation frequency of the diffusing atom. This enhances the atomic diffusion, and thus eases the diffusion bonding of two metal pieces assuming that a perfect interface contact exists.

During diffusion bonding process, coalescence of the faying surface is accomplished through the application of pressure at elevated temperature. No melting and only limited macroscopic deformation or relative motion of the parts occurs during bonding. Microscopic deformation followed by recrystallization occurs. Near the bond zone, self-diffusion in the same materials and inter-diffusion between the materials takes place simultaneously. New crystalline forms of the original elements and inter-metallic compounds may grow during the process (Kazakov *et al.*, 1985).

The success or failure of the process is decided by three variables which need a constant watch and careful adjustment. These variables are the bonding temperature, the bonding pressure (or pressing load), and the holding time (duration of pressure). The bonding temperature should be anywhere between 50% and 70% of the melting point of the most fusible materials in the composition. Elevated temperature aids the inter-diffusion of atoms across the interface of the weld, and this assists surface deformation (the crushing of surface asperities). The bonding pressure or pressing load should ensure tight contact between the edges of the pieces. It must be sufficient to aid deformation of the surface asperities and to fill all the voids in the weld zone. If the pressure is not sufficient, some of the voids will be left unfilled, and the strength of the joint will be impaired.

Diffusion bonded joints are usually characterized by scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), electron backscatter diffraction (EBSD), transmission electron microscopy (TEM) and selected area electron diffraction (SAED), high resolution TEM (HRTEM) and Fast Fourier transform (FFT) (Sónia Simões *et al.*, 2016) Various mechanical properties of the joints are also being examined.

# 2 MATERIALS AND METHODS

## 2.1 Materials

Types, grades, composition and the mechanical properties of the parent materials used in this study are as shown in Table 1 and Table 2.

Table 1: Chemical Composition and Mechanical Properties of Grey Cast Iron.

| Chemical composition (wt %) | | | | | |
|---|---|---|---|---|---|
| | Si | C | Mn | P | S |
| Grey cast iron (ASTM A48C 35) | 4.43 | 2.21 | 0.62 | 0.073 | 0.069 |
| | | | | | |
| | Tensile Strength (MPa) | | Elongation (%) | | Hardness (HB) |
| Grey cast iron (ASTM A48C 35) | 250 | | - | | 190 |

Table 2: Chemical Composition and Mechanical Properties of Low Carbon Steel.

| Chemical composition (wt %) | | | | | |
|---|---|---|---|---|---|
| | Si | C | Mn | P | S |
| Low Carbon Steel (BS Grade 250) | 0.19 | 0.10 | 0.46 | 0.011 | 0.031 |
| | | | | | |
| | Tensile Strength (MPa) | | Elongation (%) | | Hardness (HB) |
| Low Carbon Steel (BS Grade 250) | 408 | | 35 | | 195 |

## 2.2 Method

### 2.2.1 Diffusion Bonding Conditions

The main parameters that constitute common bonding conditions for diffusion bonding are namely, bonding temperature, bonding time, bonding pressure and bonding environment such as conducting of experiment under different degrees of vacuuming, shielded environment or under atmospheric pressure. For this research the first three parameters stated above are considered as variables to be investigated on their effects to the bonded joint microstructural and mechanical properties. Bonding parameter estimated variables' values were selected based on the best conditions of past experiments and most related literatures reviewed Momono, 1990., Ayob, 1990., Awfa Abdul, 2014., Kurt, 2007., Calvo, 1989).

### 2.2.2 Diffusion Bonding

Diffusion bonding of marine grades grey cast iron to low carbon steel have been conducted using a hot press machine.

### 2.2.3 Metallographic Examination

Scanning electron microscopy (SEM) and energy dispersive spectrometry (EDS) were used for a composition and weight concentration analysis of diffusion couples produced. **Tensile Strength Testing**

Few diffusion couples were subjected to a tensile test. Subsize test specimen was prepared as per ASTM standard's E8M-11. Room temperature tensile testing was carried out using a computer controlled Instron testing machine with a crosshead velocity of 0.15mm/minute.

Figure 1: Unjointed specimen (Tw=950ºC, tw=60min, Pw=10MPa).

### 2.2.5 Post Heat Treatment

Few diffusion couples were post heat treated. Temperatures selected are based on optimum recommended temperatures for diffusion welding process of the appropriate materials as this treatment is meant to allow for further diffusion process to take place. Hence the temperatures and times being chosen were $800^0$C, $900^0$C, $1000^0$C and 2, 4, 8 hours respectively.

### 2.2.6 Optical Micrograph

Photographs were taken in the vicinity of diffusion zones, along the bonding interface by optical microscope (OM).

### 2.2.7 Microhardness Testing

Micro-hardness tester of the Vickers hardness testing machine was employed. The hardness was measured across the bonding interface.

# 3 RESULTS AND DISCUSSION

## 3.1 Preliminary Study

Two couples were bonded, however both failed to result in any bonding. Figure 1 shows one of the unjointed surfaces of the diffusion couples.


Figure 2: Jointed specimen (Tw=900oC, tw=30min, Pw=10MPa).

From the investigation of the above unsuccessful bonding, further experiments were conducted with the adjustment of the equipment setting and the bonding parameters. Finally, few visually good joints were obtained with one of them shown in Figure 2.

### 3.1.1 Microstructure of Diffusion Layer

The SEM micrographs of one of the bonded diffusion couples are shown in Figure 3 taken at the interfaces of the joints.

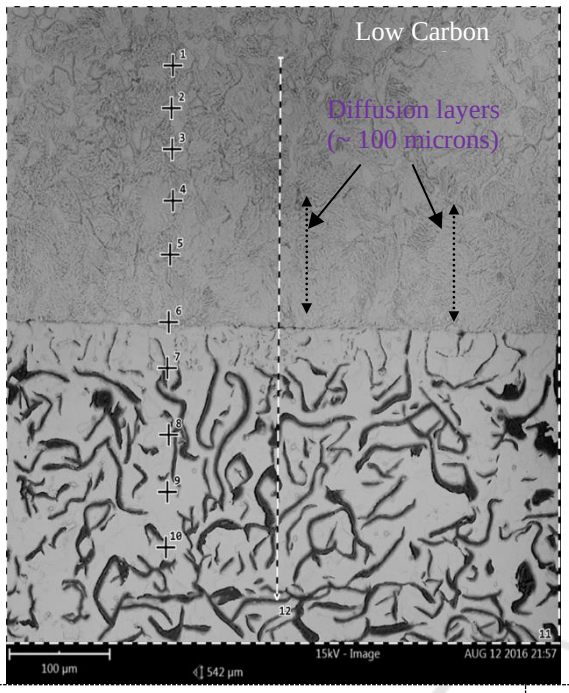



Figure 3a: SEM micrographs of grey cast iron and low carbon steel (Tw=900ºC, tw=60min, Pw=10MPa) @ x 500

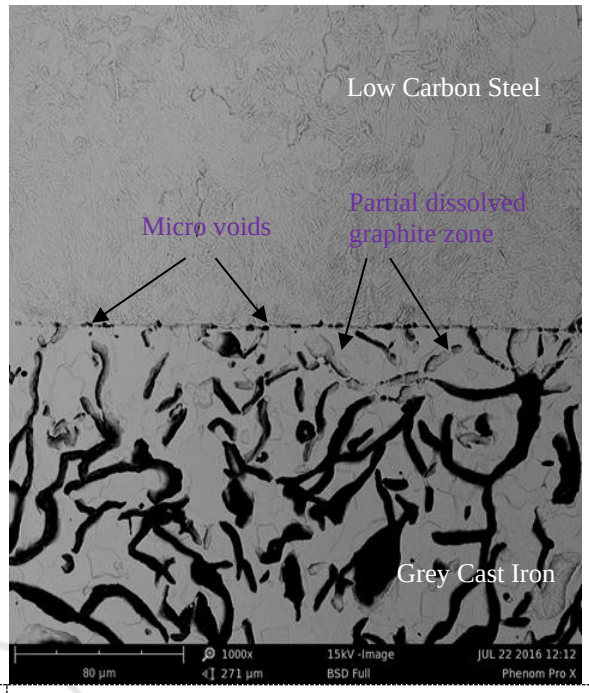



Figure 3b: SEM micrographs of grey cast iron and low carbon steel (Tw=900oC, tw=60min, Pw=10MPa) @ x 1000

The micrographs exhibit fairly good bonding along the interface of the welded joint with microvoids still present. Diffusion layer is observed on the low carbon steel side with thickness of about 100 microns measured from the bond interface as at Figure 3a. On the cast iron side close to the interface, partial dissolution of graphite flakes is visible (Figure 3b).

From the energy dispersive spectrometry (EDS) results, a rapid diffusion of carbon occurs from the cast iron to the low carbon steel. Carbon concentration is consistently high up to about 200 micron distances from the interface into the low carbon steel side (from point 6 to point 1 in Figure 3a). At point 6 (Figure 3a), silicon was traced and indicated that it has diffused into the low carbon steel as in the typical EDS graphic interface region which show changes in the weld composition as in Figure.4 and Table 3.

Table 3: EDS's Composition and Weight Concentration at point 6, of Figure 3a.

| Element | Weight Concentration |
|---|---|
| Iron | 96.5 |
| Oxygen | 0.7 |
| Carbon | 2.5 |
| Silicon | 0.3 |

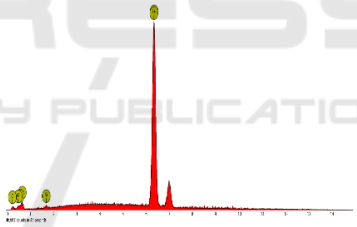


Figure 4. EDS graphic in interface region point 6, of Figure 3a

### 3.1.2 Tensile Strength of Diffusion Couple

Tensile test was conducted based on the standard DIN 50125, Type F test piece, whereby the specimens were in the original form, round bar and un-machined conditions. The results of three diffusion couples treated with the same bonding parameters except the boding time are as below.

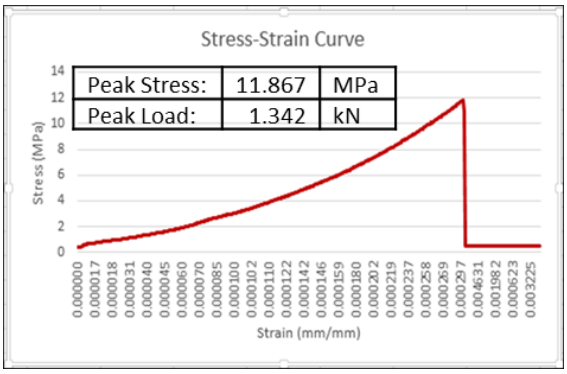



Figure 5a: True stress- strain curve of diffusion couple bonded for 1 hour.

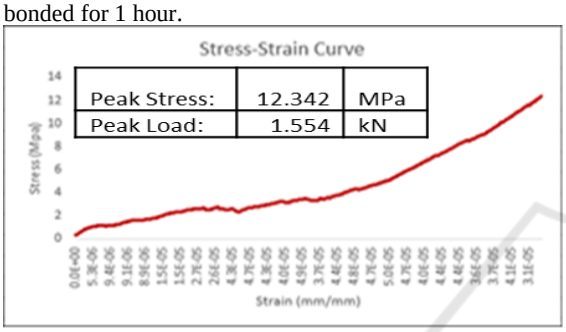



Figure 5b: True stress- strain curve of diffusion couple bonded for 2 hours.

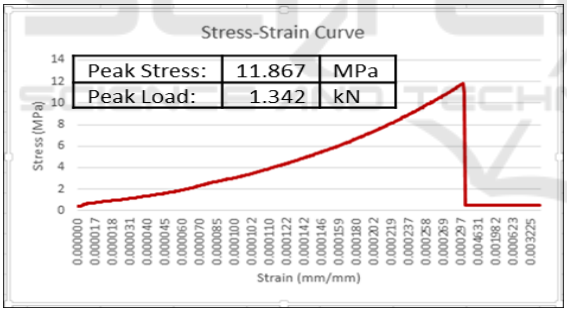



Figure 5c: True stress- strain curve of diffusion couple bonded for 4 hours.

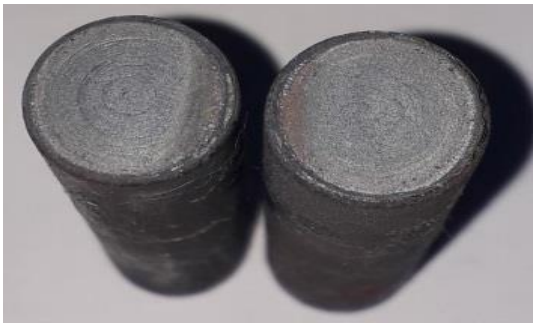


Figure 6: Fractured Surface.

From the above Figure 5 stress-strain curves result the following could be deduced:

- The strength of all the diffusion couples is very low, despite bonded.
- Effect of different bonding time on the tensile strength is not noticeable.
- As bond had still occurred, though minimal, inter diffusion of atoms across the interfaced was also found to be very minimum.
- Figure 6 shows fractured interfaces of one of the diffusion couples after the tensile test with sign of very little of inter diffusion had taken place at the edges of the specimens.

The results of these preliminary studies showed failure to produce a joint or a strong joint while the bond was incomplete. It was suspected that one of the reasons could be inadequately maintained bonding pressure arising from loss of pressure at the interfaces during bonding.

## 3.2 Post Heat Treatment

### 3.2.1 Joint's Interface Microstructure

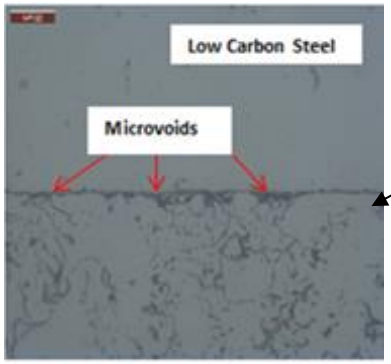
Few of the diffusion couples were subjected to heat treatment conducted under a combination of temperature of 800ºC, 900ºC and 1000ºC, and time at 2 hours, 4 hours and 8 hours respectively. Figure 7, Figure 8 and Figure 9 show the typical analysis of the interfaces and the diffusion layers taken using optical microscope (OM).

As in the Figure 7a, microvoids and interface lines are very much visible at the interfaces of the specimens treated at temperature of 800 $^0$C.

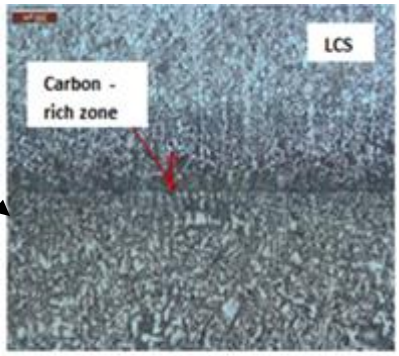
Diffusion layer of carbon rich zone has formed as observed in the Figure 7b. Spherodization or dissolution of graphite flakes is not observed on the cast iron sides close to the interface.

As in the Figure 8a, microvoids and interface lines are becoming less visible at the interfaces of the specimens. Spherodization or dissolution of graphite flakes is observed on the cast iron sides close to the interface of specimens as in Figure 8b. 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 sides respectively.

As in the Figure 9a, microvoids and interface lines almost disappear and the bond/weld also seems to be more complete. Spherodization or dissolution of graphite flakes is observed on the cast iron sides close to the interface of specimens as in Figure 9b. 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 sides




Joint interface

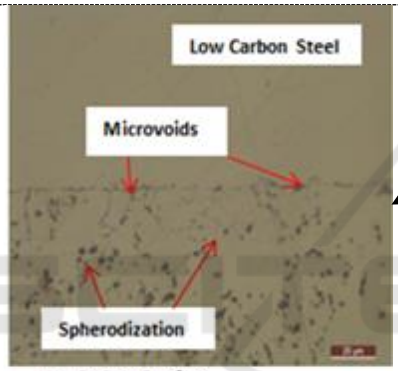



(a) Before Etching

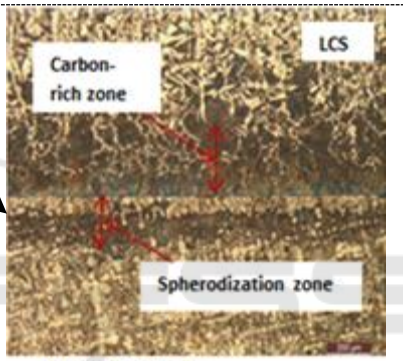
(b) After Etching

Figure 7a: OM micrographs' typical analysis of the interfaces and the diffusion layers of specimens treated at 800 $^0$C for 4 hours. Microvoids and interface lines visible

Figure 7b: OM micrographs' typical analysis of the interfaces and the diffusion layers of specimens treated at 800 $^0$C for 4 hours. Diffusion layer of carbon rich zone formed.




Joint interface

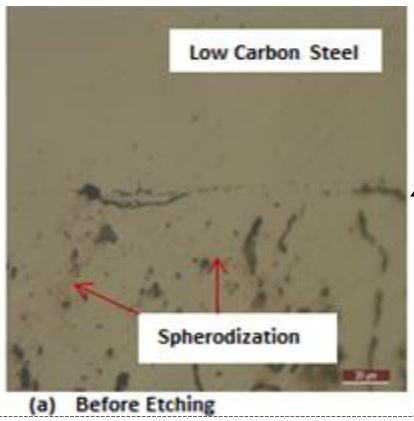



(a) Before Etching

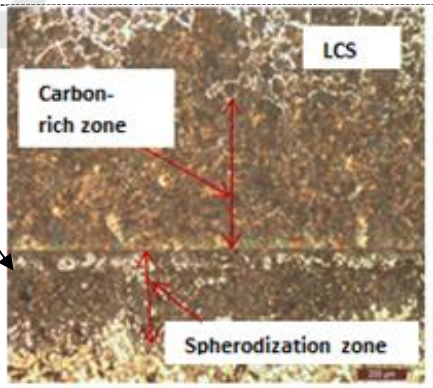
(b) After Etching

Figure 8a: OM micrographs' typical analysis of the interfaces and the diffusion layers of specimens treated at 900 $^0$C for 4 hours. Microvoids and interface lines less visible

Figure 8b: OM micrographs' typical analysis of the interfaces and the diffusion layers of specimens treated at 900 $^0$C for 4 hours. Spherodization or graphite flakes observed.




Joint interfac




(a) Before Etching

(b) After Etching

Figure 9a: OM micrographs' typical analysis of the interfaces and the diffusion layers of specimens treated at 1000 $^0$C for 4 hours. Microvoids and interface almost disappear.

Figure 9b: OM micrographs' typical analysis of the interfaces and the diffusion layers of specimens treated at 1000 $^0$C for 4 hours. Spherodization or graphite flakes observed on cast iron.

Table 4: Thickness of diffusion layers before heat treatment.

| Sample No. | Temp (ºC) | Time (hour) | Press (MPa) | | Diffusion layer thickness (microns/µm) | |
|---|---|---|---|---|---|---|
| | | | | | Carbon-rich zone | Spheroidization zone |
| 1 | 900 | 1 | 10 | | 100 | 30 |
| 2 | 950 | 0.5 | 10 | | 200 | 50 |

Table 5: Thickness of diffusion layers after heat treatment.

| Sample No. | Temp (ºC) | Time (hour) | | Diffusion layer thickness (microns/µm) | |
|---|---|---|---|---|---|
| | | | | Carbon-rich zone | Spheroidization zone |
| 1 | 800 | 2 | | 100 | 0 |
| 2 | 800 | 4 | | 200 | 0 |
| 3 | 800 | 8 | | 300 | 0 |
| 4 | 900 | 2 | | 300 | 80 |
| 5 | 900 | 4 | | 400 | 180 |
| 6 | 900 | 8 | | 500 | 300 |
| 7 | 1000 | 2 | | 650 | 200 |
| 8 | 1000 | 4 | | 750 | 300 |
| 9 | 1000 | 8 | | >1000 | >1000 |

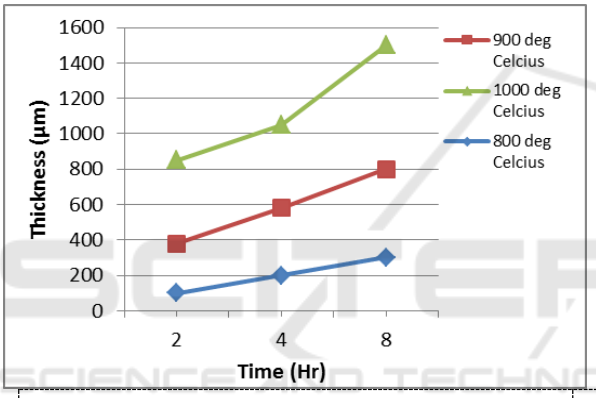



Figure 10: Time dependence of diffusion layer thickness.

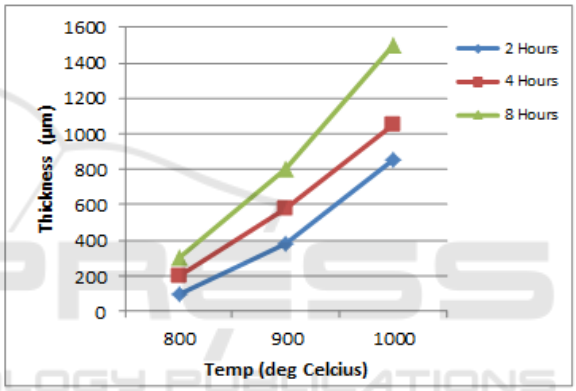



Figure 11: Temperature dependence of diffusion layer thickness

respectively. 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 (Calvo et all., 1989).

### 3.2.2 Diffusion Layer Measurement

Measurement of the diffusion layers was done directly from the OM microphotographs. Their thickness before and after heat treatment are shown in the Table.4 and Table.5 respectively. From these Table 4 and Table 5 a steady increase in thickness of diffusion layers is observed as diffusion times and temperature consecutively increase for all diffusion couples. For all heat treatment, further diffusion process has taken place with the increase of the diffusion layer thickness corresponding with increase of treatment times and temperatures. Figure 10 and Figure 11 show their correlations and interdependences.

### 3.2.3 Survey of Microhardness across Diffusion Layer

Figure 12 shows typical micro Vickers hardness gradients across the bonding interface of grey cast iron and low carbon steel of a diffusion couple. The hardness of the diffusion layer produced in all diffusion couple specimens is much higher than the hardness of the respective base metals at all treatment time and temperatures. The maximum hardness was observed close to the interface on the both sides of cast iron and low carbon steel. This corresponds to the area of diffusion layers of spheroidization zone and carbon rich zone, especially for 900ºC and 1000ºC treatments.

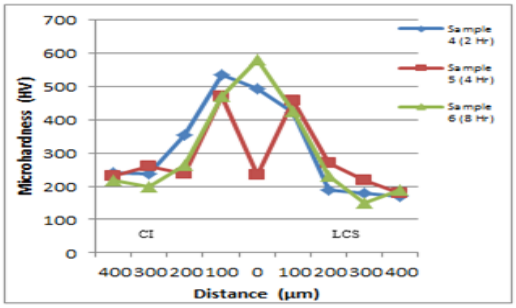



Figure 12: Microhardness profile across the interface of diffusion couple.

## 4 CONCLUSIONS

Cast iron and steel are difficult to join by the conventional fusion welding due to chemical, mechanical and structural heterogeneities associated with fusion welding. Viability of diffusion bonding based on solid state diffusion principle of cast iron to low carbon steel is predicted to be able to overcome problems faced by fusion welding, hence produce strong joint.

In earlier preliminary study a few diffusion couples of grey cast iron and low carbon steel were produced despite earlier failure. Micrograph examinations exhibit presence of microvoids along the interface of the bonded joint. The tensile strength value observed was found to be very low. Failure to produce a joint, whether a strong or complete one was suspected to be due to the loss of bonding pressure at the interfaces during bonding.

Post heat treatment improved the bond with the elimination of the interfacial voids, disappearance of the bond interface line as well as an evident complete bond which significantly is observed at a higher temperature. Structure, thickness, composition and hardness of the diffusion layer were found to be strongly influenced by the heat treatment time and temperature.

Further analysis on the bond quality will be conducted with various means of metallographic examinations and mechanical testing. Once the optimum condition has been found, investigation will be carried out to establish the diffusion mechanism, inter-diffusion coefficients and activation energy of the diffusion system.

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