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Study on the Effect of Austempering Temperature and Time on the Corrosion Resistance of Carbidic Austempered Ductile Iron (CADI) Material

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Abstract – Carbidic austempered ductile iron (CADI) has emerged as a promising engineering material to replace the ADI for higher wear resistance. It induces interest to study the corrosion behaviour of the material in chloride media, bearing in mind that equipment made from this material may be in constant contact with such media. Corrosion resistance of ductile cast iron is improved by the incorporation of an extra phase in the matrix which typically consisting of some carbides. The objective of the present work is to produce carbides in a ductile cast iron which is subsequently austempered to obtain CADI. Six variants of CADI were produced by heating carbidic ductile iron (CDI) to a austenitization temperature of 975 ºC for the period of 1 hr and quenching it in a salt bath at temperature 325 ºC and at 250 ºC for periods of 1 hr, 2 hr and 3 hr at each temperature. The corrosion resistance was evaluated by using salt spray fog test in accordance with ISO 9227 standards. The mechanical properties- bulk hardness, micro hardness evaluated and microstructure of the CADI were characterized by optical microscope and SEM. CADI sample at Austempering temperature 250 ºC and austempering time 1 hr has given best hardness 58 HRc and lesser corrosion rate 0.2094 mm/year which indicates best corrosion resistance.

Keywords – Austempering Temperature, Austempering Time, CADI, Microstructure, Optical Microscope, SEM, Salt Spray Fog Test, Corrosion Rate, Corrosion Resistance.

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INTRODUCTION

A new type of ductile iron, containing carbides immersed in the typical matrix of DI called CDI. CDI with typical ausferritic matrix is called CADI. CADI is a ductile cast iron containing carbides, that are induced either thermally or mechanically that is subsequently austempered to produce an ausferritic matrix with an engineered amount of carbides. Since its introduction in the 1970s, the usage of ADI has constantly increased. ADI material has been widely used as a structural material in the machine, automobile and agriculture industries. CADI has relatively low production costs but excellent properties, such as high tensile strength, satisfactory impact toughness and good fatigue resistance under dynamic loading conditions and excellent wear resistance [1]. CADI has attracted intensive attention in many areas such as railway, mining industry, defence structures, gears, pinions, crankshafts, centrifugal pump blades, transmissions, suspensions, earth-moving and construction equipment, etc. [2]. Corrosion rate can be measured by using different corrosion testing methods such as electrochemical test, salt spray test and total immersion test. The choice of the corrosion testing method depends upon many factors such as nature of environment, nature of exposure, type of specimen, etc. Salt spray fog test is one of the corrosion testing methods, which involves the exposure of specimens to fine spray as mist of sodium chloride solution at a specified temperature. The spray particles settle upon the test surface (which is preferably inclined) and constantly replenish and replace the film of solution on the surface [3]. The extent and nature of the corrosion of the metal or coated surface after a specified period of exposure serves as a measure of quality of corrosion. To accelerate corrosion the temperature of the media or the pH of the media or the concentration of the media can be varied [4]. The purpose of present study was to examine microstructure, assessment of mechanical properties and determining the corrosion resistance of CADI.

EXPERIMENTAL PROCEDURE

A. Material and Sample Preparation:

The pattern used in the present experiment was made of wood with standard allowances and proper finishing; the standard square casting of 15×15×200 mm was produced in the green sand mould. Six CADI samples obtained by casting were heat treated, involving an austenitizing stage of temperature 975 ºC in a muffle furnace for 1 hr, followed by an austempering in a salt bath at two different temperatures of 325 ºC and 250 ºC for different quenching times of 1 hr, 2 hr and 3 hr. Test bars were cut in two samples of dimensions 15×15×8 mm and 15×15×13 mm to test the sample for microstructural characterization and hardness measurement. As-cast sample and six CADI samples were cut to 30x10x5 mm samples by wire cut EDM. Three sets of each such sample were made for improving the accuracy by averaging the corrosion rate. Salt spray fog test was used for calculating this corrosion rate. Sample identification was done on the basis of heat treatment parameters as given in Table 1 below.

Table 1 Identification of sample according to process parameters

Sr. No.	Process Parameters	Sample ID
1	3) Austempered at 250 °C for 1 hrs.	A1
2	Austempered at 250 °C for 2 hrs.	B1
3	Austempered at 250 °C for 3 hrs.	C1
4	Austempered at 325 °C for 1 hrs.	A2
5	Austempered at 325 °C for 2 hrs.	B2
6	Austempered at 325 °C for 3 hrs.	C2
7	As-Cast-CDI	D

B. Chemical and Micro Structural Examination:

The chemical composition of CADI sample was measured by means of a spark emission optic spectrometer as shown in Table 2. Metallographic sample preparation for optical microscopy examination was conducted by using standard grinding and polishing techniques and etching with 2% Nital. After the specimens have been cut to desired shape, they were made plane by grinding on grinding wheel. After grinding was done, samples were taken for polishing by different grade silicon carbide papers - 600, 800, 1000, 1200, 1500 µm. After polishing and grinding, further polishing for getting mirror finish was done by using rotating wheel with nylon cloth mounted on it and Alumina paste for minimum 15 minutes. The images of microstructure obtained from optical microscopy were of 100X, 200X, 300X and 500 X magnifications.

Table 2 Chemical composition of As-cast CDI

Alloying element	Wt. %
C	2.19
Si	2.21
Mn	0.58
S	0.0048
P	0.0179
Cr	2.09
Cu	0.60
Ni	0.45
Ti	0.013
Mg	0.043

C. Scanning Electron Microscopy (SEM):

A scanning electron microscope (SEM) is a type of electron microscope that produces images of sample by scanning it with a focussed beam of electron. The electrons interact with atoms in the sample, producing various signals that contain information about the samples surface topography and composition. SEM Jeol Jsm-6380A was used for scanning of samples at different magnification and photographs were taken.

D. Mechanical Tests:

Rockwell hardness was measured at 150 kg load (HRc) on C-scale. A hardness profile was obtained for each alloy. In order to determine the hardness of the carbides and the matrix separately, microhardness tests were carried out by using a Vickers indenter at a 200 g load (HV200).

E. Salt Spray Fog Test:

Corrosion resistance of samples were investigated by Salt Spray Fog test. The corrosion rate was evaluated by using weight loss method as per ASTM

G1-03. The test was conducted as per ISO9227 standards. The specimens were placed in salt spray chamber for the duration of 480 hrs (21 days) as per ISO 9227 and cleaned by using mechanical method as per ISO 8407:1991.

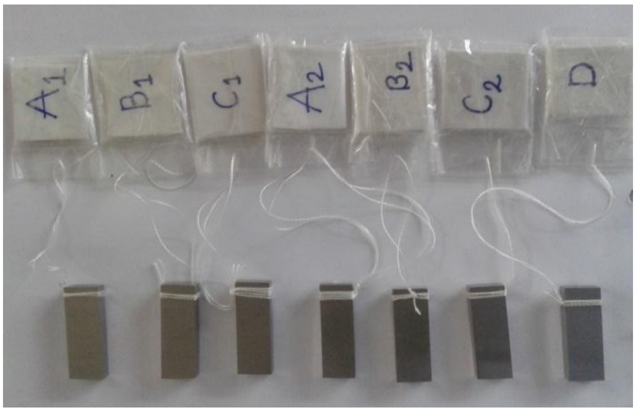

Fig.1.CADI Samples prepared for Salt Spray Fog test.

RESULTS AND DISSCUSSION

A. *Micro Structural Characterization:*

Microstructure in Fig.2.d shows white portion which are carbide traces along the grain boundary and Fig.2.b shows dark portion that is ausferrite. Microstructure Fig. 2.f shows black circular spots which are the graphite nodules. All the six microstructures show carbide traces, ausferrite and graphite nodules which indicate CADI formation.

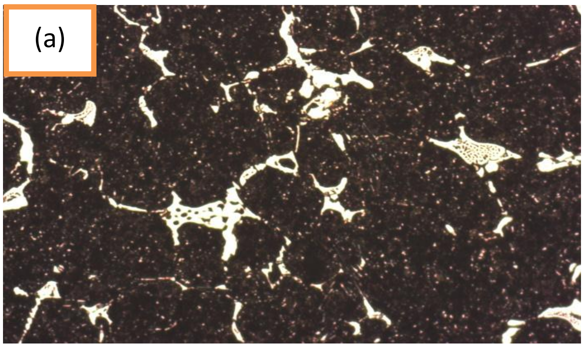

Fig.2. (a) A1-250 ^{0}C 1 hr- 200X

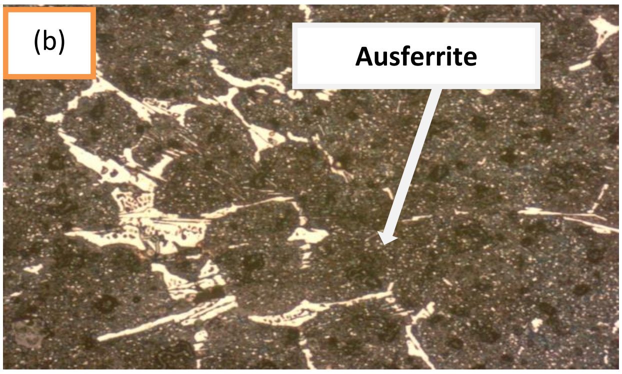

Fig.2. (b) B1-250 ^{0}C 2 hrs- 200X

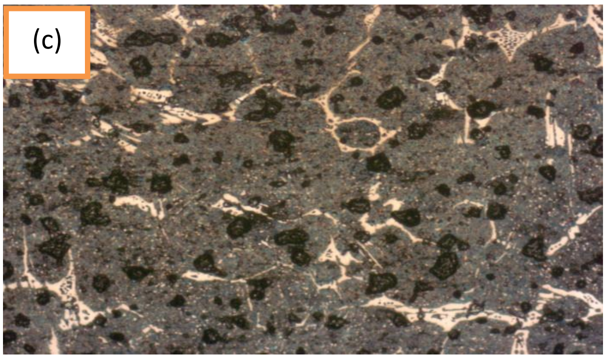

Fig.2. (c) C1-250 ^{0}C 3 hrs- 200X

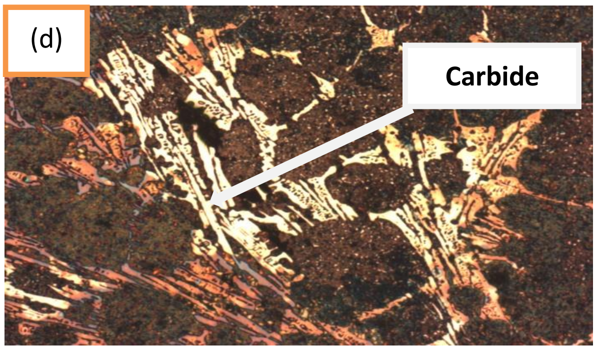

Fig.2. (d) A2-325 ^{0}C 1 hrs- 200X

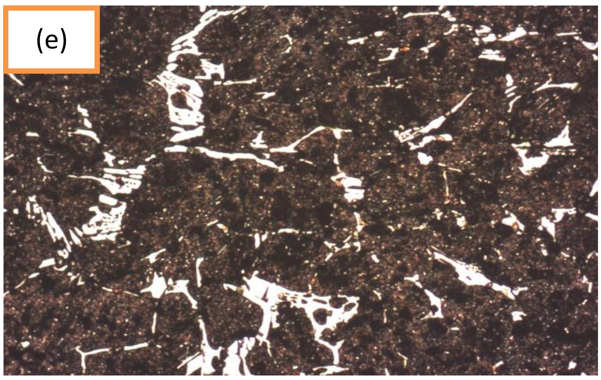

Fig.2. (e) B2-325 ^{0}C 2 hrs- 200X

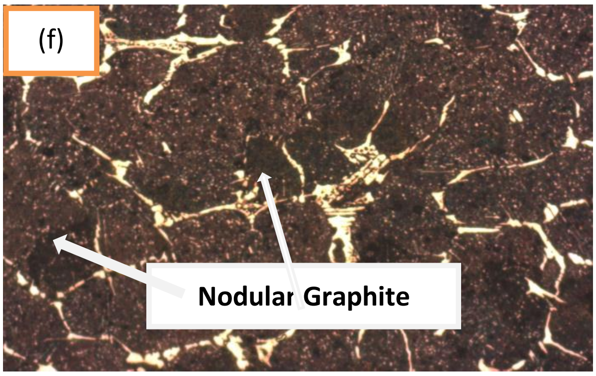

Fig.2. (f) C2- 325 ^{0}C 3 hrs- 200X

B. Scanning Electron Microscope:

After austempering CDI samples, the matrix was ausferritic, exhibiting the typical morphology i.e. reinforcement of carbide and ausferrite, which was visible in SEM.

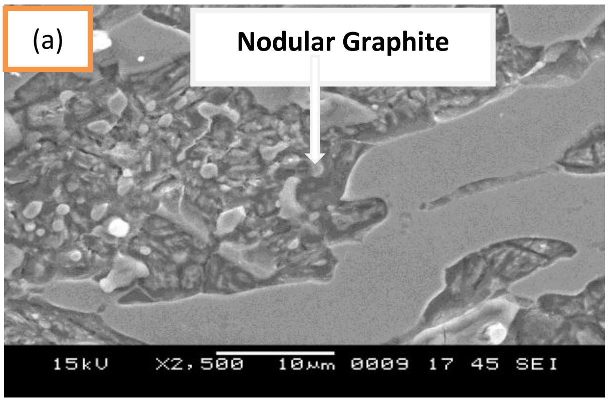

Fig. 3 (a) A1 250 ºC-1 hr- 2500X

SEM of Fig.3.b shows greyer portion which are carbides while grey portion in small pieces is retained austenite and dark needle like structure indicate ausferrite. Fig.3.a shows large amount of carbides as visible grey portion and dark spots which are graphite nodules.

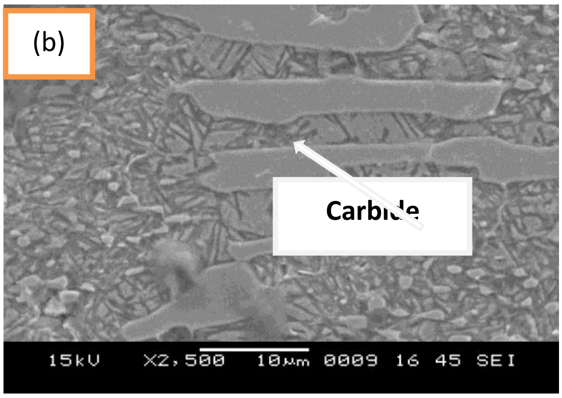

Fig. 3: (b). B1 250ºC-2 hr- 2500X

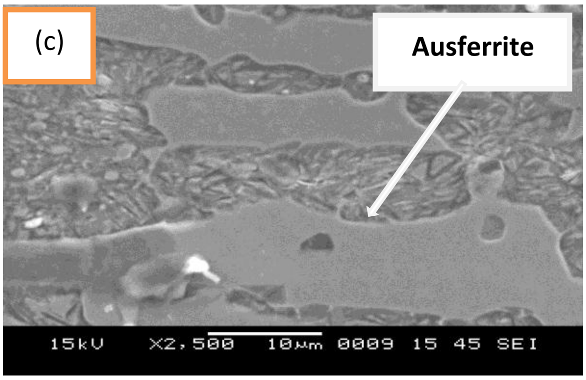

Fig. 3: (c). C1 250 ºC- 3 hr- 2500X

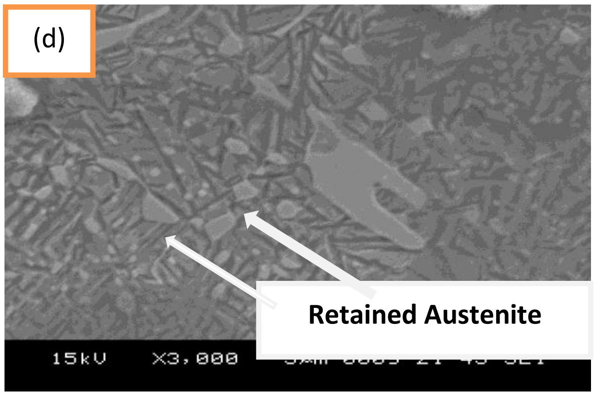

Fig. 3: (d). A2 325 ºC- 1hr- 3000X

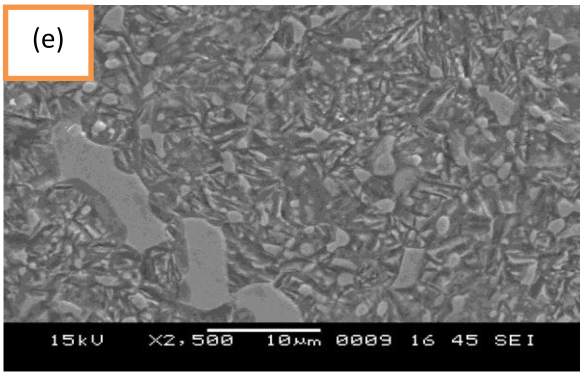

Fig. 3: (e). B2 325 ºC-2 hr- 2500X

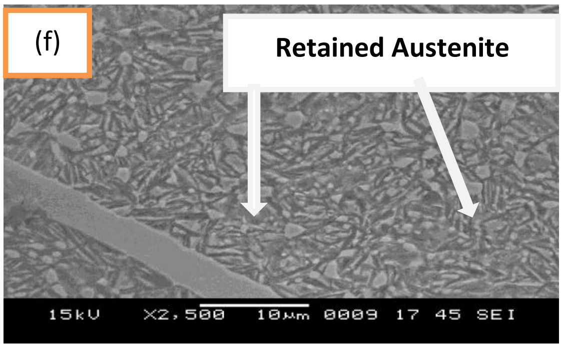

Fig. 3: (f). C2 325 ºC- 3 hr- 2500X

C. Rockwell Hardness:

The bulk hardness (HRc) was determined as an average of five measurements. Figure 4 shows that the bulk hardness values of all CADI samples are higher than as-cast. It is clearly seen that at the lower austempering temperature, 250 ºC, finer ferrite and lower amount of high carbon austenite are present, which in turn raise the hardness of the specimen. At higher austempering temperature of 325 ºC, coarser ferrite and also higher amount of high carbon austenites are present, which in turn reduce the hardness of the specimen. Retained austenite increases with decreasing austempering time at low austempering temperature of 250 °C. In other words, austenite with low carbon does not transform to bainitic ferrite and high carbon austenite.

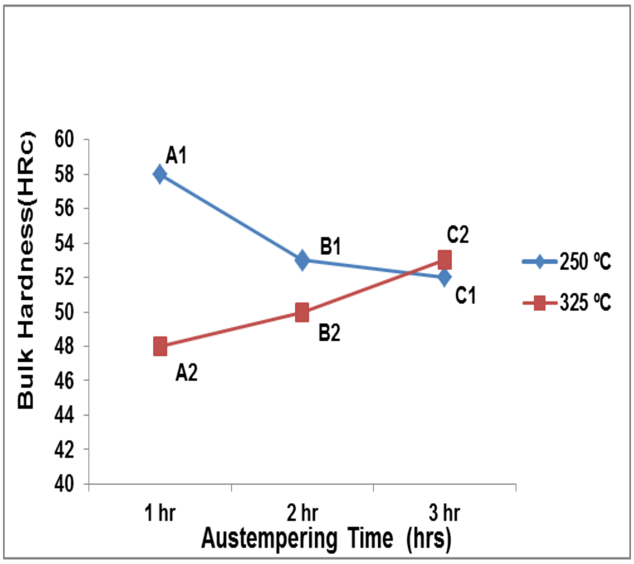

Fig. 4 Comparative graph of Bulk Hardness

Unstable austenite has been transformed to martensite and this martensite gives hardness to the microstructure. Hence the hardness for sample A1 is higher than other samples i.e. 58 HRc. whereas, at high austempering temperature (325 ºC), there is sufficient diffusion of carbon to the austenite to stabilize it, and less martensite may form during cooling to room temperature. Hence the hardness for sample A2 is lower than A1 sample (48 HRc).

D. Micro Hardness of Samples:

The Vickers microhardness was used for determining hardness. The average of five measurements was taken for each sample. Microhardness of carbide phase is found around 700HV200 to 985HV200 and for other than carbide phases, i.e. for ausferrite around 350HV200 to 550HV200. From fig. 5 it is seen that in matrix of CADI microhardness values show random change which is because carbides are randomly precipitated throughout the sample.

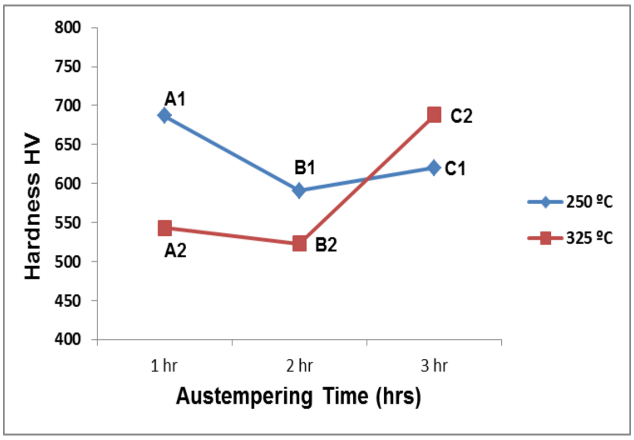

Fig. 5 Comparative graph of Micro-Hardness.

E. Corrosion Rate of CADI Samples:

Salt spray fog test is qualitative method for identifying corrosion behaviour of metals and their alloys [9]. Photographs of CADI samples, shown in fig. 6, indicate that holes and pits were present on metal surface which implies that pitting corrosion takes place. Corrosion rate of each sample was evaluated as per ASTM G1-03.

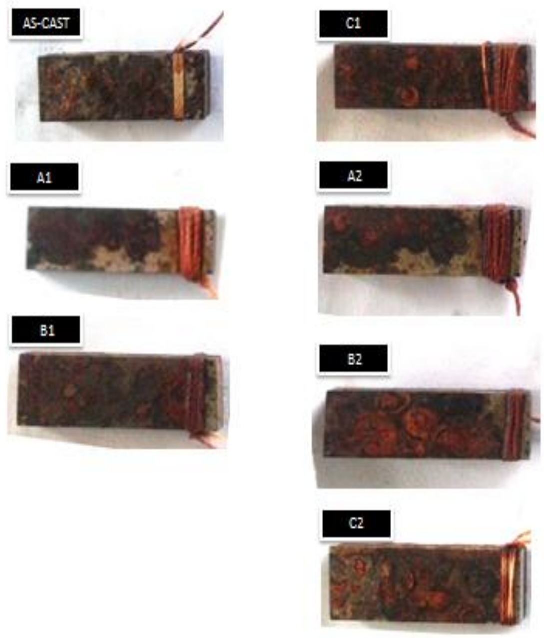

Fig.6 Corroded CADI samples.

Corrosion rate = $\frac{K \times W}{A \times T \times D}$ mm/year [10].

Where,

K = Constant = 8.76×10^4

W=Weight loss in gram

A= Corroded Surface Area, cm^2

T=Time of exposure in hrs. .

D=Density of CADI material in gm/cm^3

Table 3 Corrosion rate of CADI samples.

Sr. No	CADI Samples	Corrosion Rate (mm/year)
1	AS-CAST	0.2646
2	A1(250 ºC -1 hr)	0.2094
3	B1(250 ºC -2 hr)	0.2162
4	C1(250 ºC -3 hr)	0.2543
5	A2(325 ºC -1 hr)	0.2604
6	B2(325 ºC -2 hr)	0.2562
7	C2(325 ºC -3 hr)	0.2381

Table 3 shows the comparisons of corrosion rate of as-cast sample with CADI. Experimental results of CADI samples show same nature as that of as-cast, i.e. corrosion rate of CADI increases with increasing austempering time. Investigation shows that at lower austempering temperature (250 ºC) corrosion rate is less as compared with higher austempering temperature (325 ºC). It can be concluded that retained austenite mostly affects corrosion resistance of CADI material and their formation rate is dependent on austempering temperature and time. Fig. 7 shows that at lower austempering temperature (250 ºC), the rate of nucleation of ferrite is more, but the diffusion rate of carbon into austenite is very low which leads to the formation of fine ferrite and retained austenite. When austempering time increases from 1 to 3 hr, as shown in fig. 7, corrosion rate also increases which is because of retained austenite becoming stable.

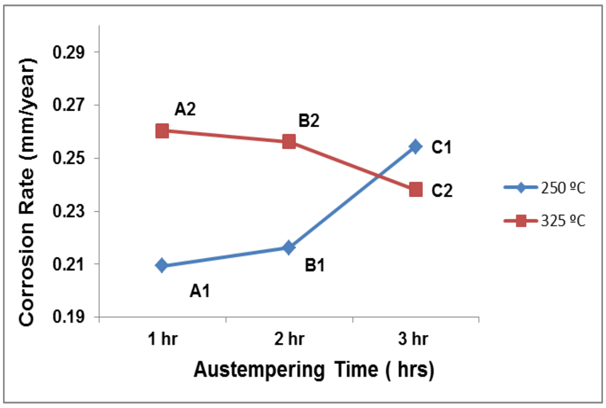

Fig.7: Austempering temperature and time effect on corrosion rate of CADI samples.

At 250 ºC temperature and 1 hr time i.e. A_1 sample has more corrosion resistance among A_1, B_1 and C_1. At higher austempering temperature (325 ºC), the austenite becomes stable at start and with increase in time again carbon starts to diffuse from austenite which forms retained austenite.

CONCLUSIONS

An attempt was made to study the effect of austempering temperature and time on corrosion resistance of CADI material. The major conclusions derived from this experimentation are:

1. It was observed in microstructure and SEM of samples that carbide, ausferrite, retained austenite and graphite nodules were present, so it can be concluded that CADI can be formed.

2. The samples austempered at higher austempering temperature had significantly higher corrosion rate which implies lesser corrosion resistance.

3. It was observed that CADI samples at lower austempering temperature of 250 ºC had more resistance to corrosion as compared with CADI samples at higher austempering temperature of 325 ºC.

4. At higher austempering temperature of 325 ºC, the corrosion resistance increases with increasing austempering time; C_2 has highest corrosion resistance among A_2, B_2 and C_2.

5. At lower austempering temperature of 250 ºC, the corrosion resistance decreases with increase in austempering time; A_1 has highest corrosion resistance among A_1, B_1, and C_1.

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