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"Experimental Assessment Of Microstructure And Corrosion Properties Of Sg Iron (400/15 Grade) Under As-Cast And Nitrotec Treated Condition"

Sadique Ali[1], Dr. Jakeer Husain[2], Dr. Amir Raza Subhani[3]

Abstract:
Spheroidal Graphite (SG) iron castings (conforming to IS 400/15 grade) are made in green sand moulds. Test specimens are then machined from the castings. One set of Specimens is then subjected to Nitrotec Treatment, treating the surface with nitrogen atmosphere which resulted in the formation of Fe_3N layer. The Nitrotec temperature selected is 570^0C and 630^0C. Specimen is held at this temperature for a fixed duration of 180 minutes and then quenched in quench bath (water based quenchant) maintained at 50^0C. The effect of Nitrotec Treatment on microstructure and corrosion is studied and compared with as-cast ones. The results of the research indicate the Graphite nodules are more or less uniform and distributed evenly in the ferritic matrix. The nodules count shows an average of 90 nodules/mm^2. Corrosion or rusting is essentially a process of oxidation in which iron combines with water and oxygen to form rust, the reddish-brown crust that forms on the surface of the iron. Nitrotec treated specimen treated at 570º C exhibit lower corrosion rate compared with the ones treated at 630º C. This may be due to the higher hardness values obtained for low Nitrotec temperature treated ones. From the above investigation it is clear that the Nitrotec treatment has an effect on the microstructure and corrosion properties of SG iron castings leading to further investigation on Nitrotec treated SG iron by changing different parameters.

Keywords: - *SG Iron, Green Sand Moulds, Nitrotec Treatment, Microstructure, Corrosion.*

1.0 Introduction:

The properties of SG iron combine the properties of cast iron and steel. Irons with varying microstructure can be obtained by carry out different heat treatments [1, 2]. The formation of nodules is achieved b[1]y the addition of nodulizers; ferrosilicon magnesium is used as a nodulizers. The matrix of the SG iron can be controlled by the base composition, by foundry practice or by subjecting the iron to heat treatment cycle [4, 5]. The process parameters play an important role and are critical in the production of SG iron as the primary concern is the formation of graphite in the form of spheroids. NITROTEC treatment belongs to a family of surface treatments carried out on metals and alloys. A newly developed treatment for Steels and Cast irons carried out in the temperature range 550ºC – 740ºC using a gaseous nitrogen bearing atmosphere [9]. The treatments develop iron nitride surface compound layers (5 and

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[1]Research Scholar, Department of Applied Sciences, Faculty of Engineering and Technology, Khaja Bandanawaz University, Kalaburagi, 585104, Karnataka, India
[2]Assistant Professor, Department of Applied Sciences, Faculty of Engineering and Technology, Khaja Bandanawaz University, Kalaburagi,585104, Karnataka, India
[3]Assistant Professor, Mechanical Engineering Department, Siwan College of Engineering and Management, Surapur, Siwan, 841226, Bihar, India

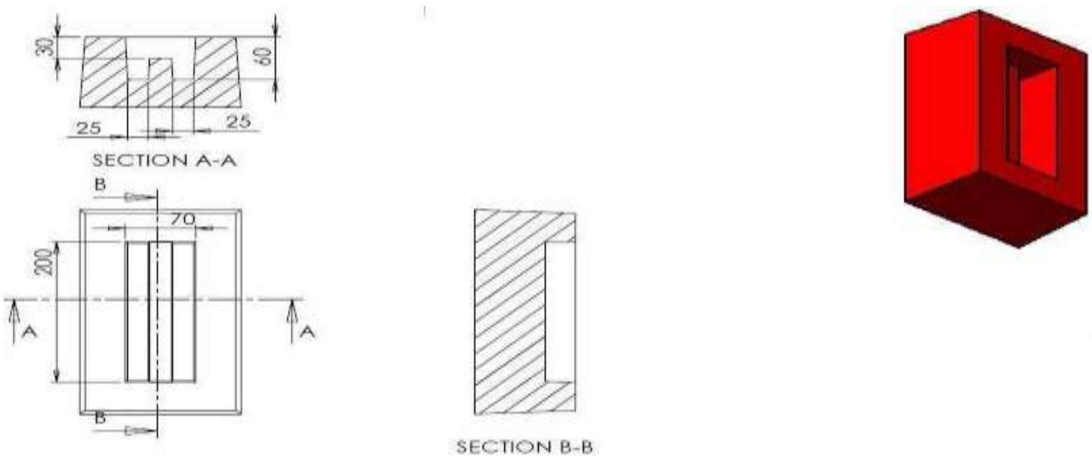
50µm thick).Corrosion or rusting is essentially a process of oxidation in which iron combines with water and oxygen to form rust, the reddish-brown crust that forms on the surface of the iron. Wear and corrosion are considered as important factor for characterizing a material. Corrosion is one of the three most commonly encountered industrial problems leading to the replacement of components and [11] assemblies in engineering. The other factors are wear and fatigue. As soon as the metals are extracted from their ores, the reverse process begins, i.e. nature tries to convert them back into the form in which they occur. This is due to the attack of the gases present in the [15] atmosphere on the surface of the metal converting it into compounds such as oxides, sulphides, carbonates sulphates etc. The effect of Nitrotec treatment on microstructure were studied and has been compared with as-cast condition, the results of the investigation are presented below.

2.0 Methodology:

Mangalore sand consisting of silica sand as base sand with angular shape and having grain fineness number of AFS 60 was used for molding [along with 12% clay and 7 % water]. Sand mix was prepared using a laboratory type of sand mixer (Capacity: 30 Kg). The properties of the sand mixture are given below: Green compression strength developed – 1050 to 1280 N/mm^2 Green sand permeability – 115 to 135. The charge consisting of pig iron, cast iron, mild steel in the form of punching and pig iron (low Sulphur content) was melted in an induction furnace. The molding, melting and casting preparation were carried out in a regular ductile iron production foundry, M/S Bangalore Metallurgical Pvt. Ltd, Hoskote, Bangalore. Melting of the charge was carried out using 3 Tons capacity, 200 Hz frequency induction furnace. The charge consisting of foundry returns, iron scrap (containing low Sulphur pig iron), steel (punching) scrap was used. The above charge materials were charged into the furnace. The furnace was switched on. When the temperature of the metal was around 1550^{o} C, desulphurization was carried out using calcium carbide (1.9% by weight basis). This was carried out to reduce the Sulphur content in the iron. This desulphurised iron was then transferred to the treatment ladle where Ferro-silicon magnesium was kept in a pocket, i.e., standard sandwich treatment method was employed. After the reaction was complete, the dross and other impurities (floating at the top of the molten metal) were skimmed off. When the temperature was around 1500^{o} C, the clean molten metal was transferred into the prepared moulds. The metal was then allowed to solidify and cool inside the sand moulds. Castings were ejected out from the moulds. Test specimens for the microstructure examination were machined as per the requirements. The above sand mixture was used for preparing the moulds. Standard square cross sectional castings of 25 mm x 25 mm x length 200mm was produced. The test samples were cut from the castings, machined and used for evaluation of different properties. Figure 1.1 shows the schematic diagram of patterns for production of ductile iron. Table 1.1 shows the composition of IS 400/15 Grade SG Iron.

Table 1: Composition of IS 400/15 Grade SG Iron.

Grade	**% C**	**% Si**	**% Mn**	**% P**	**% S**
SG 400/15	**3.69**	**2.39**	**0.30**	**0.052**	**0.010**



All dimensions are in mm

Fig. 1.1 Schematic diagram of pattern for production of ductile iron

Figure 1.2 and Figure 1.3 shows the details of melting of charge and the removal of impurities

Fig. 1.2: Melting of charge **Fig. 1.3: Dross and other impurities skimmed off**

3. Nitrotec treatment process:

The Nitrotec process encompasses a wide range of nitriding atmosphere compositions, the essential feature of which is combining ammonia (NH_3) and endothermic gas. The Nitrotec process converts the surface of the ductile iron specimen into a black, corrosion resistant iron oxide (Fe_3O_4) layer. Beneath this layer is the epsilon iron nitride (Fe_3N) layer. After the treatment, the component is quenched. The ductile iron components treated by Nitrotec process is characterized by the surface hardness. The Nitrotec Treatment was carried out in a regular heat treatment industry High Temp Furnaces, Peenya, Bangalore. Ductile iron specimen was loaded into the furnace and the temperature was increased from room temperature to the standard Nitrotec temperature. (Two different Nitrotec temperatures were selected they were namely 570^0C. The duration of the treatment was maintained constant (180 minutes)). After attaining the desired temperature, the specimen was held at that constant temperature for duration of 180 minutes (to get a surface thickness between 2 to 4 microns). The specimen was then quenched using special quenchant (water based quenchant maintained at 50^0C) for duration of 20 minutes.

The specimen for microstructure examination was machined from the as-cast ones. Tests were performed for the following conditions: As-cast condition and Nitrotec treated condition. The study of microstructure forms important part of the experiment. Microstructure helps in characterization of composition structure and properties of material. Microstructure is the structure that is observed when a polished and etched specimen of metal is viewed in an optical microscope.
Steps involved in the preparation of specimen for microstructure examination are described below: The sample for micro examination was sectioned using suitable cutter. Sharp edges, burrs and any intervening deformed material was removed by rough grinding (using an abrasive belt grinder). Grinding operation was performed in successive steps using silicon carbide, abrasive papers of 100, 220, and 400 grits. Fine grinding was done using 1500, 2500 and 3000 grit silicon carbide papers. These papers were fixed to a rotating wheel with running water to flush away loose abrasive particles. Rough polishing was performed by using distilled water on a blazed cloth fixed to the rotating wheel. Fine polishing was performed by hand on micro cloth using fine polishing compound (diamond paste). The samples are then washed in running water and dried. Samples were etched to reveal the grain structure (using 3% Nital consisting of 96-98 ml of ethanol and 2-4 ml concentrated nitric acid). After etching, sample was washed and dried thoroughly and viewed for structure examination using an optical microscope. Figure 1.4. shows the Optical microscope.

Fig. 1.4 Optical microscope

Corrosion test was carried out using a salt spray fog type apparatus conforming to the B117 specification. Flat rectangular specimen (both as-cast and Nitrotec treated) were hung from the top (inside the cabinet) were exposed to corrosive media for two different temperatures (35ºC and 45ºC). The detailed procedure of the test is given below. Figure 1.5 shows the salt spray fog type corrosion test apparatus.

Fig. 1.5: Salt spray fog type corrosion test apparatus

Test procedure: The salt solution salt spray chamber walls were cleaned thoroughly, to remove any salt residue. The temperature probes were wiped off with a damp cloth to remove any salt residue. The atomizing nozzle was cleaned with water to remove any salt residue that may plug the orifices of the nozzle. The outside of the cabinet was wiped and all the controls to minimize the chance of corrosion. The salt solution was prepared by dissolving 5 parts by weight of salt (sodium chloride) in 95 parts of the water. This salt solution was transferred into the test cabinet. Prior to the test, rectangular slab specimen of 50mm*25mm*6mm thick were weighed. The specimens were hung using a nylon wire inside the fog chamber. Specimens were exposed in a salt spray cabinet per ASTM B-117-90 using the salt solution. The outlet of the air compressor was connected to the nozzle and the heating coils were switched on; the solution gets heated up and the temperature inside the cabinet was held at 35ºC throughout the duration of the test. Compressed air at a constant pressure of $2kg/cm^2$ was blown through the nozzle for the entire duration of the test to cause bubbling action in the salt bath and create fog in the chamber. The specimen were exposed to corrosive atmosphere for 24 hours (1 day).At the end of every 24 hours, the test was stopped; specimen were removed and dipped inside the cleaning solution for 20 mins. The specimen were then removed from the solution and allowed to dry. The weight of the specimen was noted down and tabulated in a tabular column. The corrosion rate of each specimen was calculated in terms of mpy (mils per year).The test was conducted for seven days duration and the reading of initial and final weight were noted down at the end of each day of test. The graph of corrosion rate versus test duration was drawn. The above test procedure was carried out on as-cast and Nitrotec treated specimens. The above procedure was carried out for other temperature of 45 degree centigrade.

Cleaning solution:

Cleaning solution for spray fog type of test for corrosion rate measurement is given here under.(ASTM B117standard)

The chemical composition of cleaning solution is

Concentrated hydrochloric acid	1000 mL
Antimony trioxide	20gm
Stannous chloride	50gm

Formula used:
Corrosion Rate (mpy) = (534xW) / D A T
Where,
W = Weight loss in mg,
D = Density of the specimen in gm/cc,
A = Area in square mm,
T = Exposure time in hours
mpy = mils per year

4. Results and Discussion:

The results of the investigation carried out on ductile iron castings conforming to IS400/15 grade made in green sand mold for as-cast condition and Nitrotec treated condition are discussed as follows. The specimen for microstructure examination were prepared by polishing and etching using 2% nital (98% ethanol and 2% concentrated nitric acid) and viewed under optical microscope.

4.1 As-cast Condition:

It may be seen from the photo1, that the graphite nodules more or less uniform and evenly distributed. An average of 90 nodules/mm^2 is observed in the as-cast condition. Fig. 1.6 shows the Ductile Iron.

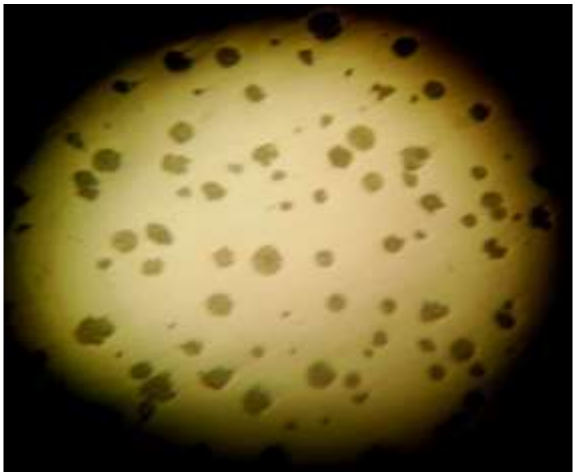
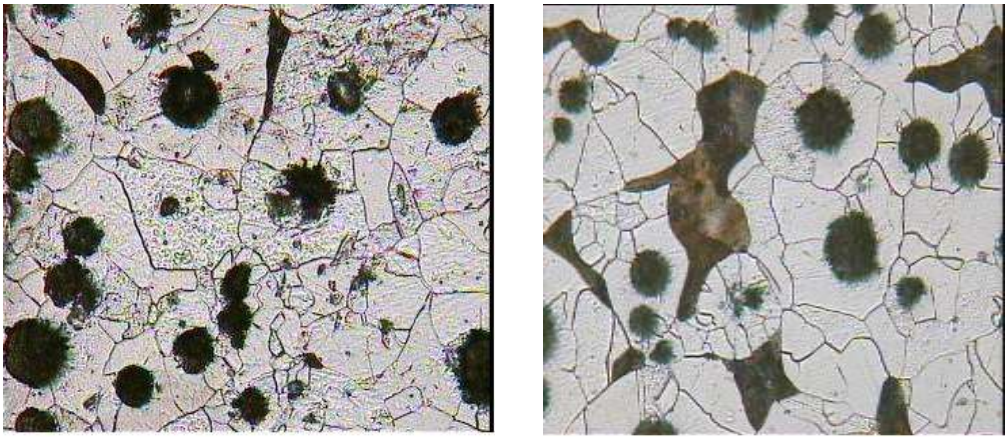


Fig. 1.6: Ductile Iron

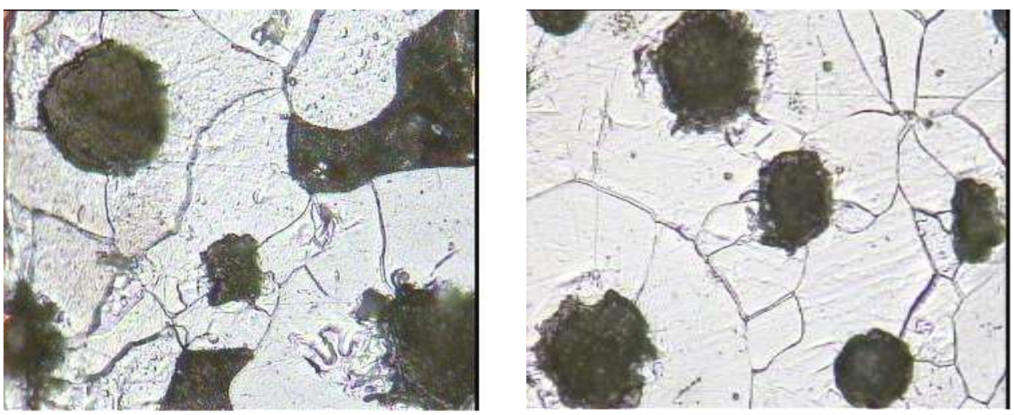
Figure 1.7 (a) shows the Ductile Iron from surface and Fig. 1.7 (b) shows the Ductile Iron towards surface.



(a) From Surface **(b) Towards Core**

Fig. 1.7: Ductile Iron

Figure 1.8 (a) shows the Ductile Iron from surface and Fig. 1.8 (b) shows the Ductile Iron towards surface.



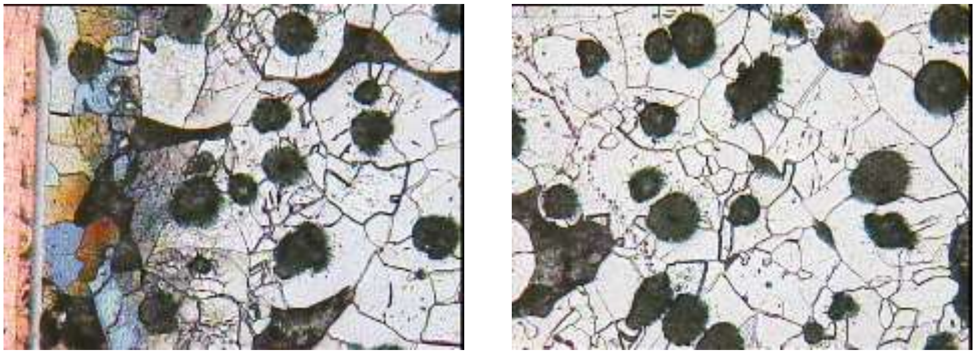
(a) From Surface **(b) Towards Core**

Fig. 1.8: Ductile Iron

4.2 Nitrotec Treated Condition:

Figure 1.9 (a) shows the Ductile Iron from surface and Fig. 1.9 (b) shows the Ductile Iron towards surface.

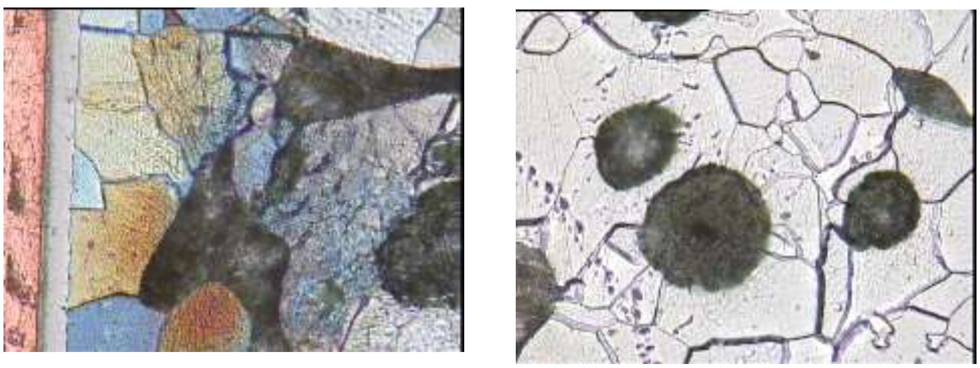
From the Figure 1.9 and Figure 1.10, it can be seen that specimen subjected to Nitrotec treatment results in a Compound layer consisting of iron oxide (Fe_3O_4) and nitride (Fe_3N) layer of 4 to 5 microns thick is observed. Surface hardness observed is in the range of 635 to 672 VHN.



(a) From Surface **(b) Towards Core**

Fig. 1. 9: Ductile Iron

Figure 1.10 (a) shows the Ductile Iron from surface and Fig. 1.10 (b) shows the Ductile Iron towards surface.



(a) From Surface **(b) Towards Core**

Fig. 1. 10: Ductile Iron

4.3 Corrosion Tests:

The results of the corrosion tests carried out on as-cast ductile iron specimen (conforming toIS400/15 grade) and Nitrotec treated specimen treated for two different temperatures (570^0 C and 630^0C for 180 minutes' duration) are presented under the following. Fig. 1.11 shows the Variation of corrosion rate (mpy) with time for the as-cast specimen and Nitrotec treated specimen.

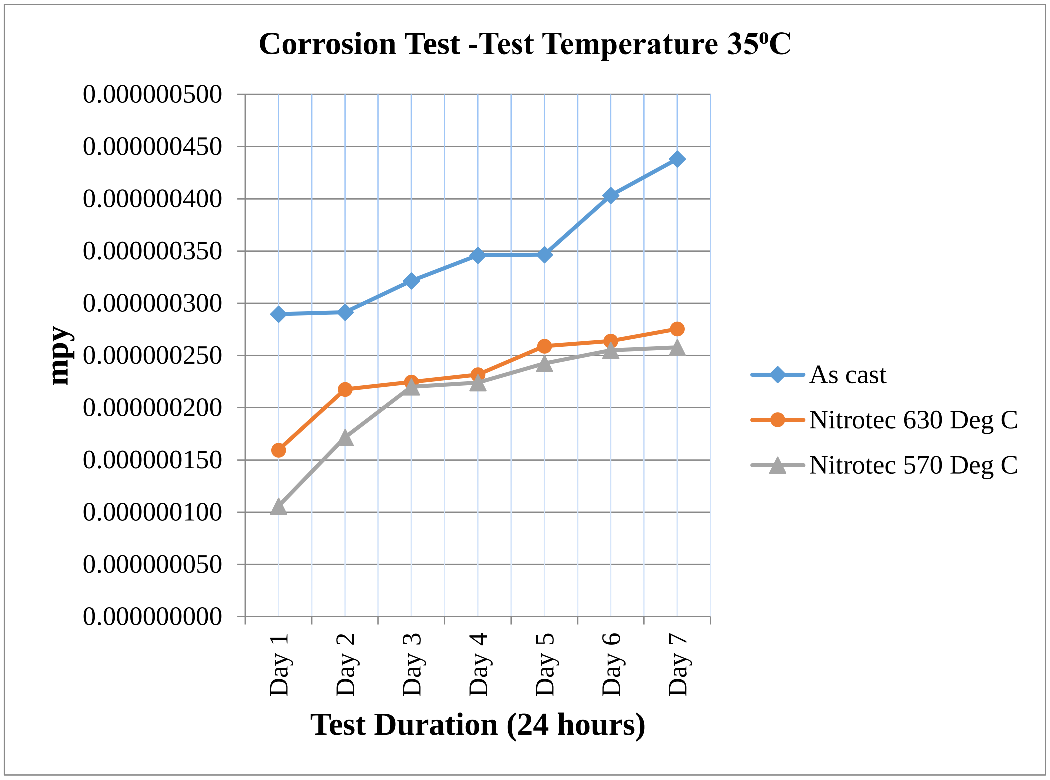


Fig. 1.11 Variation of corrosion rate (mpy) with time for the as-cast specimen and Nitrotec treated specimen

Figure 1.12 shows the Variation of corrosion rate (mpy) with time for the as-cast specimen and Nitrotec treated specimen.

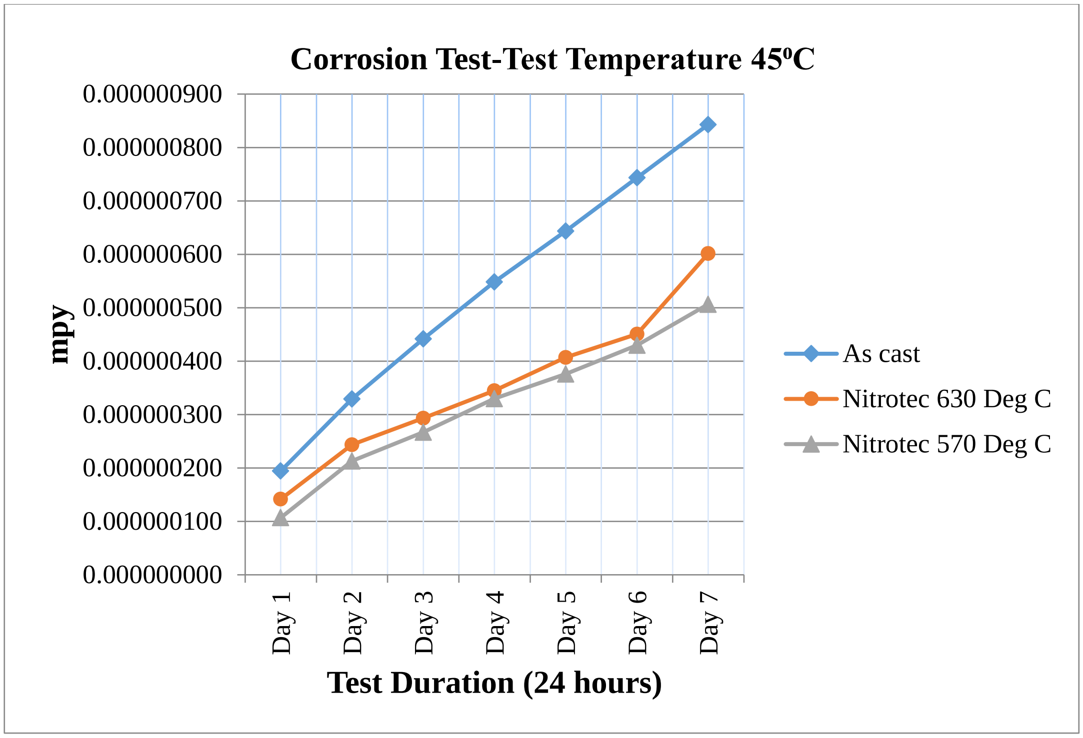


Fig. 1.12 Variation of corrosion rate (mpy) with time for the as-cast specimen and Nitrotec treated specimen

It can be observed from these Figures 1.11 and 1.12 that:

- Corrosion rate increases steadily with increase in the time of testing.
- Severe corrosion is noticed in the ascast condition - whereas less wear is seen with Nitrotec treated specimen.
- Nitrotec treated specimen treated at 570º C exhibit lower corrosion rate compared with the ones treated at 630º C. This may be due to the higher hardness values obtained for low Nitrotec temperature treated ones.
- Also, the corrosion rate exhibited by specimen for different conditions seems to be less when treated at 35 ℃ test temperature.
- Hence, the study clearly indicates that Nitrotec treatment has a significant effect on the corrosion properties of ductile iron

5. Conclusion:

From the studies carried out on as-cast ductile iron and Nitrotec treated ductile iron, the following observations can be made:

Graphite nodules are more or less uniform in size and are evenly distributed in ferritic matrix. As-cast ductile iron exhibits ferritic matrix. Upon subjecting the specimen to Nitrotec Treatment, a hard black layer of corrosion resistant iron oxide (Fe_3O_4) layer is formed. Beneath this layer is the epsilon iron nitride (Fe_3N) layer is clearly seen. From the corrosion tests carried out on as-cast ductile iron and ductile iron subjected to Nitrotec treatment, it is observed that: Corrosion rate increases steadily with increase in the time of testing. Severe corrosion is noticed in the as-cast condition - whereas less corrosion is seen with Nitrotec treated specimen. Nitrotec treated specimen treated at 570º C exhibit lower corrosion rate compared with the ones treated at 630º C. This may be due to the higher hardness values obtained for low Nitrotec temperature treated ones.

Hence from the above studies carried out on as-cast ductile iron and Nitrotec treated ductile iron, it is clear that upon Nitrotec treatment of ductile iron specimen exhibits better hardness values. It results in superior wear resistance properties. It has also significant effect on the corrosion properties of ductile iron. These in turn can increase the potential applications of the ductile iron.

A review of papers indicates that substantial information relating to the structure and mechanical properties of SG iron is available. Also substantial information is available relating to the Nitrotec treatment on steels and cast iron. However, there is a scope for conducting studies to evaluate mechanical properties, wear properties and corrosion properties on the Nitrotec treated SG iron.

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