

# Study of Effect of Etching Time and Concentration on Microstructure of SG 400/12 Grade Ductile Cast Iron

Nilesh T. Khot\*, Anil S. Morkane\*, Umesh V. Borkar\*, Sanket S. Maske\*, Prof. B. S. Kamble\*

\*UG students at Department of Production Engineering, KIT's College of Engg., Kolhapur, Maharashtra, India

# Assistant Professor at Dept. of Production Engg., KIT's College of Engg., Kolhapur, Maharashtra, India

## ABSTRACT

The study of material microstructure is known as Metallography. This is very critical in estimating the material properties. Metallography can be a very useful quality control tool too. By examining the micrographs one can determine the quality of the product. Micro structural examination is generally done on a sample whose surface has been grinded or polished to a mirror-like finish. SG cast iron is characterized by nodules-like graphite distribution when seen under an optical microscope. The as-polished samples are also etched with various etchants to look at the underlying phases (e.g. ferrite, pearlite, cementite), which also have an effect on the properties of the SG cast-iron. If a casting has predominant ferrite phase, then it would be soft. Ample presence of carbide will lead to a hard material. The study reported here deals with the etching of SG cast iron (400/12 Grade) samples for short etching time (4 – 10 sec) and also for long etching time (30 sec and 60 sec). For the purpose of observing the microstructure we have used Scanning Electron Microscope (SEM). The optimal solution is taken by the use of regression analysis technique. Etching is the selective corrosion of the surface under consideration to reveal the underlying features. The purpose of etching is to reveal certain features (e.g. grain boundary and phases) which are not visible in the as-polished condition.

**Keywords :** Sampeling, Polishing, Etching, Microscopic examination, Nital, Picral

## I. INTRODUCTION

Metallography has been described as both a science and an art. Traditionally, metallography has been the study of the microscopic structure of metals and alloys using optical metallographs, electron microscopes or other surface analysis equipment. It can be more precisely defined as the scientific discipline of observing and determining the chemical and atomic structure and spatial distribution of the constituents, inclusions or phases in metallic alloys. The surface of a metallographic specimen is prepared by various methods of grinding, polishing, and etching. After preparation, it is often analysed using optical or

electron microscopy. Using only metallographic techniques, a skilled technician can identify alloys, predict material properties and its performance and reliability. Thus metallography is used in materials development, incoming inspection, production and manufacturing control, and for failure analysis; in other words, product reliability.

## II. LITERATURE REVIEW

Chakraborty et.al (1) have studied, the most commonly used concentrations of Nital and Picral and has found that with Etching for 10 sec, 2% Nital provides an optimum etch. For extended etching, Picral provides

an optimum etch at 70 sec of etching which can go up to 80 sec of etch and the concentration of Picral may be between 3% - 4%. David A. Scott (3) had observed that the grey cast iron contains 4% carbon and a little phosphorus in early 20th century. Before etching, the outlines of some of the thick graphite. T.M. Hegazy (4) has undergone study of effect of NaOH concentration and etching duration on some properties of  $\gamma$ -irradiated PADC and it is revealed that bulk etching rate shows weak dependence on the etching time. David P. B. Samuelso (6) had used digital image correlation technique (DIC). This method is mostly used for determination of strains on a macroscopic level e.g. testing of a tensile test piece or any other test piece or component that is exposed to a load. And it is found that it is possible to investigate strain-fields on a microscopic scale, different phases of cast irons and their strain response during loading and to investigate the effect of eutectic cells and primary austenite on the strain distribution. Donald Zipperian (7) has enlisted different types of etchants used for etching are mentioned. George Vander Voort (21) has studied the microstructure of CI using different etchants namely Nital and Picral at various concentration and time rates. Also discussed about the how to prepare the sample specimen for metallographic study without damage.

### III. EXPERIMENTAL PROCEDURE



**Figure 1.** Test Speciman of SG Iron grade 400/12

The SG Iron material of grade 400/12 is taken as sample under study. The initial material which in the form of rod cut into the length 10 \* Ø15 mm as shown in Figure with the help Semi-automatic Hack Saw Machine.



**Figure 2.** Speciman samples cut of SG Iron grade 400/12 to carry out buffing operation

The surface on which the polishing operations have to be done, initially they are made free from burs and sharp edges with the help of file. Manual polishing with successively 100, 200, 300 and 400 grade sand papers are used. After the semi finishing of the specimens now they are prepared for actual metallography. To achieve the mirror like surface finish they are placed on the Buffing Machine.



**Figure 3.** Buffing Machine

For this experimental study, the most commonly used Nitric acid and Picric acid were used as etchants. Nital and Picral with 2% and 4% concentration were used with acid dipping dwell period of 4,10,30,60 seconds period. The 2% Nital was prepared by adding 2 ml of concentrated Nitric acid ( $\text{HNO}_3$ ) in 100 ml of 99% Ethanol. Similarly 3% and 4% Picral were prepared by adding 3 ml and 4 ml of picric acid in 100 ml 99%

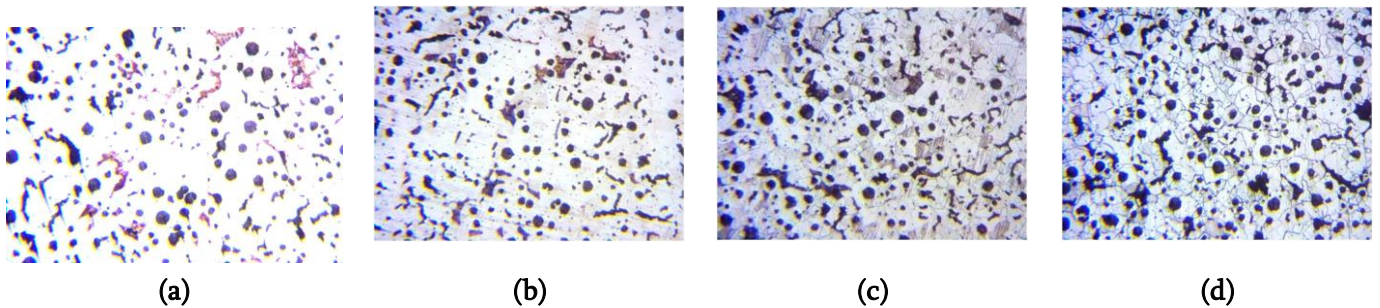
ethanol respectively. Every time freshly prepared etchant were used for each study.

**Table .1** Experimental parameters and combination

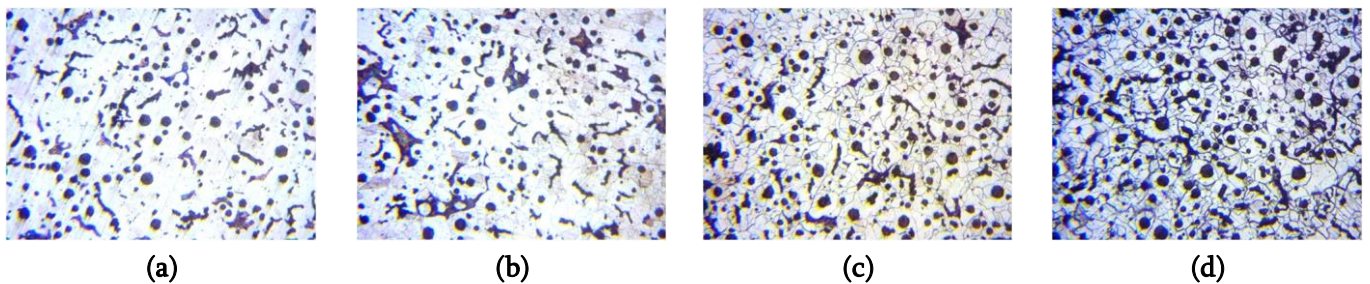
Exp.No.	Etchant	Etchant Concentration	Etching Time (Sec)
1	Nital	2%	4
2			10
3			30
4			60
5	Nital	4%	4
6			10
7			30
8			60

9	Picral	2%	4
10			10
11			30
12			60
13	Picral	4%	4
14			10
15			30
16			60

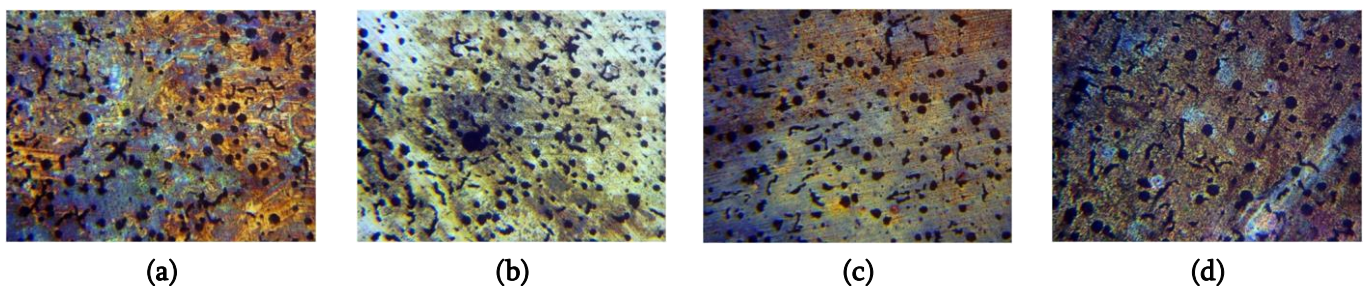
After the preparation of specimen, these are dipped in the etchants of various concentrations. Then the microstructural images are observed under SEM (Scanning Electron Microscope).



**Figure 4.** Samples etched with 2%Nital with 4sec, 10 sec, 30 sec and 60Sec dwell time period with magnification 100X

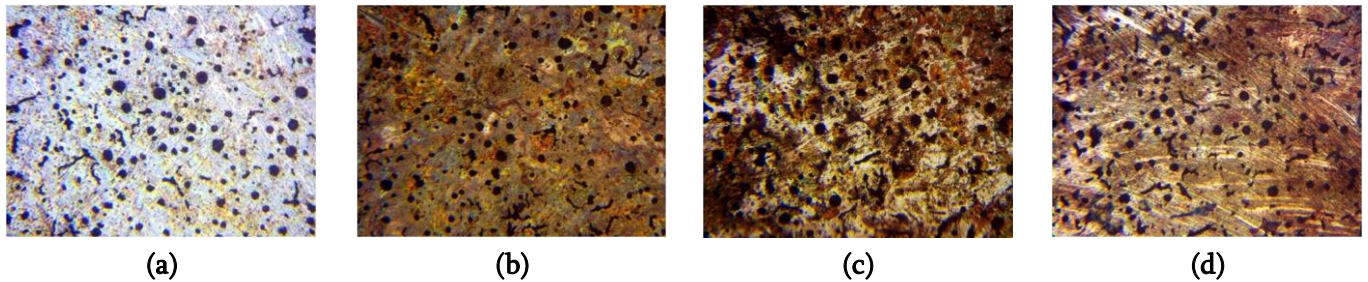


**Figure 5.** Samples etched with 4%Nital and with 4sec, 10 sec, 30 sec and 60Sec dwell time period with magnification 100X



**Figure 6.** Samples etched with 2%Picral and with 4sec, 10 sec, 30 sec and 60Sec dwell time period with magnification 100X





**Figure 7. Samples etched with 4%Picral and with 4sec, 10 sec, 30 sec and 60Sec dwell time period with magnification 100X**

**Table .2 Percentage of Nodularity and nodules observed under SEM**

Etchant	Etching Time (sec)	% Nodularity	Nodules (%)	Etchant	Etching Time (sec)	% Nodularity	Nodules (%)
2% Nital	4	84	11.2	2% Picral	4	82	9.95
	10	80	10.3		10	88	9.95
	30	85	9.5		30	82	9.95
	60	83	8.1		60	87	9.8
4% Nital	4	79	10.2	4% Picral	4	86	8.9
	10	83	8.1		10	87	8.8
	30	82	9.8		30	85	10.5
	60	80	9.1		60	87	10.1

#### IV. MICROSTRUCTURAL OBSERVATIONS

The magnified images from the etched samples with 2% Nitric acid are as shown in figure 4. It is clearly observed changes in the pearlite and Ferritic grain boundry visibility with an increase in the etching time from 4 sec to 60 sec. Figure 4a clearly shows signs of Insufficient etching concentration with no any clear visibility of phase as well as Ferritic grain boundry. With increase in etching holding time the microstructural contrast also increase. With 2% Nital concentration and etching holding time of 4 to 60 sec., an average of 9.7% Nodules are observed .Similarly magnified images from the etched samples with 4% Nitric acid are as shown in figure 5. Here also it is observed that there is increased contrast with increase in acid concentration and etching holding time. An average of 9.3% Nodules are observed with 4% Nital concentration and time of 4 to 60 sec. With 4% Nital

concentration and time of 30 and 60 sec there is clear visibility of Ferritic grain boundry. There is clear observation of Pearlitic matrix with 9.40% pearlite & 82.50% Ferrite.

The magnified images from the etched samples with 2% Picric acid are as shown in figure 6. There is no sign of visibility of Ternary phosphorous eutectic (carbides) in structure. With increase in etching holding time there is visible Contrast among the different phase with increased darkness. With 2 and 4% Nitric and Picric acid Ternary phosphorous eutectic (carbides) are not observed in structure.

#### V. CONCLUSION

Shorter period etching duration of 4 sec and 10 sec with 2% Nitric acid visualises clear phases and

nodules. With extended etching duration period of 30 sec with 4% Nitric acid gives optimum and clear microstructure visibility. Picric acid provides better results with 4% and 4 sec etching duration time.

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