CHAPTER 4

EXPERIMENTAL DETAILS

In this study, laser boriding experiments have been carried out on nickel and chromium electroplated AISI 1020 steel. Hence, the experimental details regarding the following are described:

1. Details about the base material (substrate).

2. Equipment and chemicals used for continuous pack boriding and interrupted pack boriding.

3. Equipment and chemicals used for nickel and chromium electroplating and laser boriding (called multicomponent laser boriding).

4. Details about the structural characterisation and properties evaluation to study the different boride layers resulting from continuous pack boriding, interrupted pack boriding and multicomponent laser boriding.

4.1 BASE MATERIAL

Today, steel is one of the most common materials in the world, with more than 1.3 billions tons produced annually. It is a major component in buildings, infrastructure, tools, ships, automobiles, machines, appliances and weapons. Carbon steel is the most common form of steel as its price is relatively low while it provides material properties that are acceptable for
many applications. Low/medium carbon steel has relatively high toughness (combination of ductility and strength) as compared with high carbon steels. Hence, it was selected as the base material.

4.1.1 Chemical Composition

The chemical composition of the carbon steel (AISI 1020) used in this study is given in the Table 4.1. The chemical composition was checked using a Baird Spectrovac.

Table 4.1 Chemical composition (in wt. %) of the base material

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Ni</th>
<th>Al</th>
<th>B</th>
<th>Mo</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.230</td>
<td>0.061</td>
<td>0.330</td>
<td>0.018</td>
<td>0.042</td>
<td>0.008</td>
<td>0.003</td>
<td>0.019</td>
<td>Bal</td>
</tr>
</tbody>
</table>

4.1.2 Specimen Preparation for Boriding

The steel specimens were machined to the dimensions according to type of characterisation. The specimen dimensions for optical, microhardness, XRD and fracture toughness studies were 20 mm (length) x 20 mm (breadth) x 5 mm (thickness). The specimen dimensions for the charpy impact test were 55 mm (length) x 10 mm (breadth) x 10 mm (thickness). The specimen dimensions for wear and corrosion study was 10 mm in diameter and 31 mm height. The surfaces of the specimens were ground up to 3/0 emery paper just before each boriding process. Acetone was used for degreasing the specimens.

4.2 CONTINUOUS PACK BORIDING

The continuous pack boriding is a conventional thermo-chemical boriding process. The paste method was selected. The paste was prepared using 30% boron carbide (B₄C), 50% silicon carbide (SiC), 10% potassium
fluroborate (KBF₄), 5% sodium carbonate (Na₂CO₃) and 5% titanium dioxide (TiO₂). Sodium silicate (Na₂SiO₃) was used as a binder. The binder and water ratio was approximately 2:1. The paste was applied on the steel surface manually. The paste thickness was approximately 1 mm. The specimens were dried at room temperature for 2 h and then it was dried in an oven at a temperature of 120 °C for 2 h. The specimens were placed inside the air tight sealed stainless steel box. The pack inside the stainless steel box contains SiC. Continuous pack boriding was carried out on AISI 1020 steel specimens in a muffle furnace at 950 °C for 3 h. The muffle furnace used for continuous pack boriding is as shown in the Figure 4.1.

![Muffle furnace used for continuous pack boriding and interrupted pack boriding](image)

**Figure 4.1** Muffle furnace used for continuous pack boriding and interrupted pack boriding

### 4.3 INTERRUPTED PACK BORIDING

Interrupted pack boriding is a modified pack boriding process. The packing procedure is similar to continuous pack boriding, but the difference is
in the heat treatment cycle of the process. The specimens were subjected to heating at 950 °C for 1 h. After 1 h of boriding, the stainless steel box containing the boriding mixture and specimens were removed from the furnace and allowed to cool in still air for 45 min and then it was loaded in the muffle furnace as shown in the Figure 4.1. This procedure was repeated three times.

4.4 MULTICOMPONENT (Ni + Cr + B₄C) LASER BORIDING

This is a three stage process. First Ni and then Cr were electroplated on the surface of the AISI 1020 steel as two different layers one over the other. Then a paste of B₄C was applied on the as-plated surface and subsequently laser boriding was done at different energy densities. Argon was used as a shielding gas to prevent oxidation of material during laser boriding. The laser boriding experiments were carried out at WRI, BHEL, Tiruchirapalli, Tamilnadu, India.

4.4.1 Equipments for Electroplating

Welded mild steel tank was used as electroplating vat. The thickness of the steel plate is 5 mm. A low-voltage direct current (dc) source is required for electroplating. For this purpose a dynamo, motor generator or rectifier has been employed. The current from the rectifier is supplied to the electroplating vat by means of bus bars. These are copper rods or flexible wires for amperages up to 150 A and copper or tin-plated aluminium for higher amperages. A 6 cm² cross-section copper bar is used to pass 1000 A. The bus bars must be positioned in appropriate insulators. To enable suspension of the anodes and the jobs, copper or brass rods or tubes are positioned over the tank by means of porcelain supporters. The suspender bars are connected to the bus bars with cast iron bronze fittings. A moving-coil
type ammeter and voltmeter are used to measure the current and voltage respectively.

### 4.4.2 Chemicals and Process Parameters for Electroplating

The chemicals and process parameters for nickel and chromium electroplating are listed in the following Table 4.2.

**Table 4.2 Chemicals and process parameters for nickel and chromium electroplating**

<table>
<thead>
<tr>
<th>Chemicals used and Plating parameters</th>
<th>Nickel Electroplating</th>
<th>Chromium Electroplating</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anode</td>
<td>Ni anode</td>
<td>Lead-Tin anode (93% Pb-7% Sn)</td>
</tr>
<tr>
<td>Electrolyte</td>
<td>Nickel metal 60-80 g/l</td>
<td>Chromium sulphate 20 g/l</td>
</tr>
<tr>
<td></td>
<td>Nickel Chloride 55-60 g/l</td>
<td>Potassium sulphate 150 g/l</td>
</tr>
<tr>
<td></td>
<td>Nickel sulphate 200-300 g/l</td>
<td>Sodium sulphate 100 g/l</td>
</tr>
<tr>
<td></td>
<td>Boric acid 35-45 g/l</td>
<td>Boric acid 60 g/l</td>
</tr>
<tr>
<td></td>
<td>PH 4.8-5.5</td>
<td>PH 3.5-3.7</td>
</tr>
<tr>
<td></td>
<td>Temperature 35 °C – 40 °C</td>
<td>Temperature 45 °C – 55 °C</td>
</tr>
<tr>
<td>Voltage</td>
<td>5-6 V</td>
<td>4-10 V</td>
</tr>
<tr>
<td>Current density</td>
<td>16 A/dm² (0.16 A/cm²)</td>
<td>4-10 A/dm² (0.04 – 0.10 A/cm²)</td>
</tr>
<tr>
<td>Rate of deposition</td>
<td>10 µm/ hour</td>
<td>12 µm/ hour</td>
</tr>
</tbody>
</table>

The process sequences for nickel electroplating followed by chromium electroplating are given in the Figure 4.2.
Figure 4.2 Flowchart for nickel and chromium electroplating process sequence

Ni & Cr Electroplating Process Flow Chart

Degreasing in kerosene

Saw dust cleaning

Caustic wash (NaOH flakes mixed with water)

Water wash

Acid cleaning (50% HCL and 50% water) Buffing

Anodic cleaning

Water wash

10% HCL etching

Water washes 1 & 2

5% H₂SO₄ acid

Ni plating

DM water wash

Chrome plating
4.4.3 Optical Microstructure of Ni and Cr Electroplated AISI 1020 Steel

The optical microstructure of Ni and Cr electroplated AISI 1020 steel is as shown in the Figure 4.3.

![Optical microstructure of Ni and Cr electroplated AISI 1020 steel](image)

**Figure 4.3** Optical microstructure of Ni and Cr electroplated AISI 1020 steel

The thickness of nickel and chromium layers were approximately 44 - 52 μm and 18 - 22 μm respectively.

4.4.4 Equipment for Laser Boriding

A complete laser material processing system consists of a laser source (Nd: YAG laser), a beam guidance system (either fixed or flexible) to deliver the laser beam to the processing head. A central CNC unit to control all the functions of the system like operation of the laser, adjustment of the laser power, scanning speed and other parameters. Figure 4.4 shows the schematic of a laser surface alloying system.
4.4.5 Laser Boriding Process

The B₄C powder supplied by Boron Carbide (India) Limited was used as the boron source for laser boriding process. The B₄C powder contains approximately 80% boron and 20% carbon. Poly vinyl alcohol (PVA) was used as the binder. The binder and water ratio was approximately 2:1. The boron carbide (B₄C) paste was applied on the as-plated steel surface. The thickness of the paste was about 1 mm. The specimens were dried at room temperature for 2 h and then it was dried in an oven at a temperature of 120 °C for 2 h. Laser boriding was carried out using 2 kW continuous wave Nd: YAG laser of wave length, λ =1.064 nm. During the operation, laser beam head was held stationary and the specimen was moved to and fro. The process was carried out at different energy densities (E) by keeping constant laser power at 500W, at different scanning speeds such as 1.25, 1.00, 0.75,
0.50, 0.40 and 0.30 m/min and at two different spot diameters 1.5 x 10^{-3} m and 1.62 x 10^{-3} m. The energy densities of the laser beam were varied from 19 x 10^6 J/m² to 85 x 10^6 J/m². The energy density of the laser beam is expressed in terms of laser beam power, scanning speed and spot diameter of the laser beam.

\[
\text{Energy density (J/m}^2\text{)} = \frac{4P}{\pi D^2} \times \frac{D}{V} = \frac{4P}{\pi DV}
\]

The energy density was calculated using the equation (4.1).

The laser boriding process parameters are given in the Table 4.3.

**Table 4.3 Laser boriding parameters used in the present research**

<table>
<thead>
<tr>
<th>S. No</th>
<th>Power (P) in W</th>
<th>Scanning speed (V) in m/min</th>
<th>Spot diameter of beam (D) x 10^{-3} m</th>
<th>Energy Density (E) x 10^6 J/m²</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>500</td>
<td>1.25</td>
<td>1.62</td>
<td>19</td>
</tr>
<tr>
<td>2</td>
<td>500</td>
<td>1.00</td>
<td>1.62</td>
<td>24</td>
</tr>
<tr>
<td>3</td>
<td>500</td>
<td>0.75</td>
<td>1.62</td>
<td>31</td>
</tr>
<tr>
<td>4</td>
<td>500</td>
<td>0.50</td>
<td>1.62</td>
<td>47</td>
</tr>
<tr>
<td>5</td>
<td>500</td>
<td>0.40</td>
<td>1.62</td>
<td>59</td>
</tr>
<tr>
<td>6</td>
<td>500</td>
<td>0.40</td>
<td>1.50</td>
<td>64</td>
</tr>
<tr>
<td>7</td>
<td>500</td>
<td>0.30</td>
<td>1.62</td>
<td>79</td>
</tr>
<tr>
<td>8</td>
<td>500</td>
<td>0.30</td>
<td>1.50</td>
<td>85</td>
</tr>
</tbody>
</table>

The focal length was 0.3 m. The overlap between successive passes was about 33%. Argon gas was supplied at the rate of 25 lit/min to avoid oxidation.
4.5 STRUCTURE EVALUATION

4.5.1 Optical Microstructural Observations

The continuously pack borided and interrupted pack borided and multicomponent laser borided specimens were rubbed mechanically on an emery paper (No.600) to remove the particles sticking to the surface. Then the specimens were ground very gently on one side only to remove the very hard boride layer using a pedestal grinder. After grinding to this level, the specimen was mounted using a cold mounting material, ground on rough emery papers (No. 320 -600) to get a flat surface and then on finer emery papers from 1/0 to 4/0 sequentially. Then the specimen was washed and polished on velvet cloth using diamond paste and kerosene. The polished specimen was cleaned in running water, swabbed with cotton and dried. Then it was etched with 3% Nital to reveal the boride layer. The microstructure was observed and photographed by the Zeiss Axiovert optical microscope as shown in the Figure 4.5.

Figure 4.5 Zeiss Axiovert optical microscope
4.5.1.1 Optical microstructural observations

The continuous pack borided, interrupted pack borided and multicomponent laser borided (treated at different energy densities) specimens were studied by optical microscope. For optical microstructural observations, all the photos were taken at 200X magnification.

4.5.1.2 Boride layer thickness measurements

The boride layer thicknesses of continuous pack borided, interrupted pack borided and multicomponent laser borided layers were measured at ten different positions and averaged.

For measuring case thickness, the optical photos were taken at 200X magnification for continuously pack borided, interrupted pack borided specimens and 50X magnification for multicomponent laser borided specimens.

4.5.2 Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS)

The metallographically polished specimens were studied using JEOL JSM 6360 SEM as shown in the Figure 4.6 at suitable magnification. EDS analyses were carried out using INCA Penta FET-x3, OXFORD available at Karunya University, Coimbatore, India. It has a powerful new Si (Li) detector, which has 30 mm² detecting crystal, with the ability to detect elements down to beryllium with no compromise in analytical performance.

The following specimens were analysed:

2. The amounts of B, Cr, Ni, Fe and C in the laser borided layer and transition zone using INCA Penta FET-x3 EDS system.


Figure 4.6 JEOL JSM 6360 Scanning electron microscope

4.5.3 Phase Analyses Using X-Ray Diffraction (XRD) Technique

The phase analyses of multicomponent laser borided specimens treated at different energy densities were evaluated by XRD (SHIMADZU) as shown in the Figure 4.7 operating at 30 mA and 30 kV with CoK$_\alpha$ radiation with $\lambda$ =1.78897 A$^\circ$. The set scanning speed and scanning range are 5 $^\circ$/min and 10 – 80 $^\circ$ respectively.
4.6 PROPERTIES EVALUATION

4.6.1 Microhardness Measurements

Microhardness measurements were carried out using a Mitutoyo Microhardness tester as shown in Figure 4.8. For this study only 50 grams load was used. The microhardness measurements of different boride coatings such as continuously pack borided, interrupted pack borided and multicomponent laser borided specimens were carried out along the depth of the boride layer. At any selected distance from the surface, measurements were taken at three locations and the average value was taken. Such measurements were done at various depths from the surface to the core as shown in the Figure 4.9.
Figure 4.8 Mitutoyo microhardness tester

Figure 4.9 Schematic representation of microhardness measurement along the depth of boride layer

4.6.2 Ductile/Brittle Fracture Analyses Using Charpy Impact Test

Impact tests were conducted using charpy impact testing machine as shown in Figure 4.10. The standard method for impact testing with V-notch
charpy test was slightly modified by having a boride layer on one face opposite to the notch. The dimensions of the material used for this test was 55 mm length, 10 mm breadth and 10 mm thickness. The width and depth of V-shaped notch was 2 mm with an angle of 45° respectively. AISI 1020 steel, continuously pack borided, interrupted pack borided and multicomponent laser borided specimens were tested to measure the charpy impact toughness in joules. The impact tests were performed on 3 specimens and all the values were taken. The fractured surface was analysed using macrophotography and scanning electron microscopy. By observing the fractured surfaces from macrophotographs, the percentage of ductile shear areas in the continuously pack borided, interrupted pack borided and multicomponent laser borided specimens was measured by comparing fractured surface of AISI 1020 steel as a reference.

![Figure 4.10 Charpy Impact testing machine](image)
4.6.3 Vickers Microindentation Fracture Toughness Test (VMIF)

Fracture toughness ($K_c$) of the continuously pack borided, interrupted pack borided and multicomponent laser borided specimens were estimated by micro-Vickers indentations using Vickers microhardness testers namely Mitutoyo (Figure 4.8) and Zwick (Figure 4.11) on polished cross-sections employing ASTM E384 standard.

Figure 4.11 Zwick microhardness tester

Fifty grams load was used to measure the hardness of continuous pack borided, interrupted pack borided and multicomponent laser borided layers. The schematic model representing Vickers microindentation showing crack length ($2c$) is as shown in the Figure 4.12 (Uslu et al 2007).
Figure 4.12 Schematic model representing Vickers microindentation showing crack

The fracture toughness was calculated from the equation (4.3).

\[
K_c = 0.028 \left( \frac{E}{H} \right)^{1/2} \frac{P}{c^{3/2}} 
\]  

(4.3)

Half crack length (c) was measured from optical microcopy. Different loads in the range of 1 N to 68.6 N were applied on the boride layers to produce cracks. The load at which cracks appear was estimated by trial and error method.

4.6.4 Evaluation of Wear Loss

Wear tests were carried out under dry sliding conditions on a DUCOM pin-on-disc universal tribometer model TR-20-M-106 as shown in Figure 4.13. The wear tests were conducted as per the ASTM G99-05 standard. Different borided AISI 1020 steel specimens such as continuously pack borided, interrupted pack borided and multicomponent laser borided were used as pins. The diameter of the pin was 8 mm. The contact surfaces of the pins were flat and cylindrical. The counterface material (disc) was made of continuously pack borided austenitic stainless steel of dimensions -
diameter 90 mm and thickness 6 mm. The boride layer thickness was around 28 – 33 µm. The microhardness of the disc was around 1923 - 1936 HV. The pins were made to slide on the borided steel disc at 1 m/s sliding speed under the load of 50 N for 1000 m sliding distance. The wear tests were conducted at room temperature. Before and after the test, coated pins were ultrasonically cleaned, dried and weighed using an electronic analytical balance to an accuracy of 0.01 mg. By using the data acquisition system, the frictional force was recorded automatically with respect to time. The coefficient of friction was calculated by dividing the frictional force by applied load. Wear losses of AISI 1020 steel and different boride coatings such as continuous, interrupted and multi component laser borided (treated at different energy densities) were calculated based on mass loss of the specimens.

Figure 4.13 DUCOM TR-20-M-106 pin-on-disc tribometer

4.6.5 Evaluation of Corrosion Rate

Specimens were pressed into a Teflon holder and one side of the specimen was exposed to the corrosive environment. 2.6 grams of cupric
chloride dehydrate (CuCl$_2$.2H$_2$O) in glacial acetic acid (CH$_3$COOH) was used to study the chemical stability of AISI 1020 steel, continuous pack borided, interrupted pack borided and multicomponent laser borided layers. The potentiodynamic polarization studies were carried out in a conventional three-electrode electrochemical cell.

Specimens of working area 0.785 cm$^2$ were used as the working electrode (WE). A saturated calomel electrode (SCE) was used as the reference electrode, while the platinum leaf was used as counter electrode (CE). Prior to each experiment, the specimens were polished mechanically using 220, 400, 600, 800 and 1200 silicon carbide emery sheets, rinsed with distilled water and then degreased with acetone. An electrochemical system (PARSTAT 2273 Potentiostat/Galvanostat) as shown in Figure 4.14 developed by Princeton Applied Research, USA was used to run the tests and the PowerSuite software (version 3.21) was used to collect and evaluate the experimental data.

![Figure 4.14 PARSTAT 2273 Potentiostat/Galvanostat used for corrosion study](image)