1.0 PURPOSE

The purpose of this document is to outline the general procedure to be followed for chemical etching of metallographic samples.

2.0 SCOPE

This document provides an overview of chemical etching and must be read by any employee, student or researcher required to carry out chemical etching of samples.

Specific Safety Guide Data Sheets have been prepared for individual chemical etchants and are to be used as standard procedures for making up and using those solutions, refer Appendices I and II.

3.0 REFERENCES

- Appendices I & II: Macro and Micro Etchants
- MA-MS3-SF-01: Safety Manual
- MSDS: Material Safety Data Sheets
- SP-MS3-04-07: Electrolytic Polishing
- ASM Metals Handbook: Metallography & Microstructures
- Metallographic Etching: G Petzow
- AS2576-1982: Welding Consumables for Build-Up and Wear Resistance
- Typical Microstructures of Cast Metals - The Institute of British Foundrymen
5.0 PROCEDURE

5.1 SAFETY ASPECTS

Many of the chemicals used and chemical mixtures prepared for the purpose of etching samples for metallographic examination are highly flammable, corrosive and/or toxic.

Safety as well as being a management responsibility is also a personal responsibility. The final responsibility rests with the individual. Therefore it is each persons responsibility to become familiar with the safe handling, storage and disposal of chemicals used in this section.

This procedure is not intended to cover the safety aspects of preparing and using chemicals in the section. Current Safe Work Procedures have been prepared for the etchants, referred to in Appendices I & II and only suitably trained persons will prepare etchants. Other relevant safety information is also available. See section 3 of this document.

Before any person carries out any chemical etching of samples they must have read the following documentation:

a) This Standard Procedure

b) The Discipline Safety Manual

c) The Materials Safety Data Sheets on the chemicals to be handled.

d) The Safe Work Procedure on the preparation of the selected chemical etchant.

On occasions where a person may be required to use an etchant for which no Safe work procedure is available, they must seek permission from their supervisor to do so. The supervisor must then ensure that appropriate reference material, covering any hazards in preparing and using the new etchant, has been sourced and read before permitting the etchant to be made up and used.

If appropriate a Safe work procedure should be drafted for future use.
It should be noted that an “Emergency Shower” and “Eye Wash Station” are available adjacent to the fume cupboards.

5.2 BASIC THEORY

Etching is a metallographic technique which is necessary to reveal grain size, microstructure and general homogeneity of metals and alloys.

The theory of etching polished surfaces is based on different phases and/or different grain orientation having different solubilities and consequently etching at different rates. The less soluble phases are left in relief while the more soluble are dissolved away.

Etching may be divided into two main classes - micro and macro.

Micro-etching is utilised for microscopic examination at magnifications above 10X while macro-etching is for magnification below 10X. Macro-etching involves considerably higher rates of metal removal than does micro-etching.

5.3 MICRO-ETCHING

Micro-etching is carried out by immersing the sample in the etchant selected as being appropriate for that metal or alloy (see Appendices) for a time which can be determined only from experience. However, as a general rule the sample is immersed until such time as the original highly reflective surface becomes cloudy or milky.

The general procedure for micro-etching is summarised below.

Clean the polished sample of its polishing abrasive.

Either swab sample with cotton wool saturated with etchant or immerse sample in etchant. Watch sample closely and when polished surface appears cloudy rinse with cold water.

Dry sample immediately by rinsing with alcohol and drying with compressed air or the hot air dryer.

**NOTE:** If condensation occurs, causing spotting or pitting, heat the sample under a hot water stream from the tap and re-dry with alcohol and compressed air.

Sample should be etched as soon as possible after polishing to avoid formation of passive oxide layers which impede the etching reaction.
5.4 MACRO-ETCHING

Macro-etching is carried out by immersing a sample in a bath of strong reagents, usually strong mineral acids (see Appendices). It is often necessary to heat the bath to hasten the etching process. Prior sample preparation is not as critical as for micro-etching due to the greater depth and severity of the etching operation. Generally a "Snow" ground surface finish is sufficient.

The sample is left in the acid bath for the required time (up to 20 minutes or so), then removed and washed under running water. It may be necessary to scrub the surface with a stiff brush to remove etching debris from the surface.

The sample is then dried by rinsing with alcohol and drying with compressed air. To preserve the etch, the surface may be sprayed with transparent acrylic lacquer.

Macro-etching is useful for revealing surface seams, internal cracks and porosity. Other features revealed include segregation, dendritic patterns, grain size, decarburisation, carburisation, depth of rim zone and heat affected zones.

6.0 REFERENCE PERSONNEL LIST

Prior to amending this standard procedure, refer proposed amendments to:

Principal Metallurgist Product Investigations

7.0 DOCUMENTATION

Appendix 1  -  Macro-etchants & Associated Safety Guide Data Sheet Reference
Appendix 2  -  Micro-etchants & Associated Safety Guide Data Sheet Reference
# APPENDIX I

## MACRO ETCHANTS

<table>
<thead>
<tr>
<th>Uses</th>
<th>Etchant</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Iron &amp; Steel</td>
<td>Nital 5% or 10%</td>
<td>Routine general etchant. Reveals as-cast structure of castings, carburisation or decarb, segregation hardened zones.</td>
</tr>
<tr>
<td>Iron &amp; Steel</td>
<td>Mixed Acids</td>
<td>Good general etchant. Use hot or cold. Heat increases reaction rate.</td>
</tr>
<tr>
<td>Iron &amp; Steel</td>
<td>Dilute HCl</td>
<td>Reveals porosity, inclusions, hardened zones, segregation. Heat increases reaction rate.</td>
</tr>
<tr>
<td>Iron &amp; Steel</td>
<td>Fry’s Reagent</td>
<td>Reveals flow lines</td>
</tr>
<tr>
<td>Iron &amp; Steel</td>
<td>Chemical Polish</td>
<td>Reveals columnar boundaries in cast slab samples.</td>
</tr>
<tr>
<td>Iron &amp; Steel</td>
<td>Oberhoffer’s Reagent</td>
<td>Reveals segregation of common alloying elements.</td>
</tr>
<tr>
<td>Copper &amp; Copper Alloys</td>
<td>Acid Ferric Chloride</td>
<td>Gives good contrast and reveals dendrites in cast structures.</td>
</tr>
</tbody>
</table>
### MICRO ETCHANTS

<table>
<thead>
<tr>
<th>Uses</th>
<th>Etchant</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Iron &amp; Steel</td>
<td>Nital 3% or 5%</td>
<td>Most common etchant used. Reveals ferrite grain boundaries, pearlite, martensite and bainite. Not as good as Picral for high resolution work with low temperature transformation products.</td>
</tr>
<tr>
<td>Iron &amp; Steel</td>
<td>Picral</td>
<td>As above but more sensitive to carbide than other features. Good for revealing carbides in cold rolled and annealed strip. Gives superior resolution with fine pearlite, martensite and bainite.</td>
</tr>
<tr>
<td>Iron &amp; Steel</td>
<td>Brauners Reagent</td>
<td>Reveals austenite grain boundaries in tempered steels.</td>
</tr>
<tr>
<td>Iron &amp; Steel</td>
<td>Fry’s Reagent</td>
<td>Reveals deformed regions and flow lines. Temper at 150 to 200°C prior to etching.</td>
</tr>
<tr>
<td>Iron &amp; Steel</td>
<td>SASPA</td>
<td>Segregation sensitive etchant. Also good for flow lines.</td>
</tr>
<tr>
<td>Iron &amp; Steel</td>
<td>LePera’s Reagent</td>
<td>Good for quantitative assessment of martensite, bainite in dual phase steels. Martensite appears white, bainite appears black and ferrite appears tan.</td>
</tr>
<tr>
<td>Stainless &amp; Alloy Steels</td>
<td>Aqua Regia</td>
<td>Reveals general structure of stainless steels.</td>
</tr>
<tr>
<td>Stainless &amp; Alloy Steels</td>
<td>Glycer Regia</td>
<td>For austenitic iron-chromium based alloys.</td>
</tr>
<tr>
<td>Stainless &amp; Alloy Steels</td>
<td>Acid Ferric Chloride</td>
<td>General stainless steel etchant.</td>
</tr>
<tr>
<td>Stainless &amp; Alloy Steels</td>
<td>Vilella’s Reagent</td>
<td>Etches Fe-Cr, Fe-Ni, Fe-Cr-Mn steels. Use hot to reveal austenite grain boundaries.</td>
</tr>
<tr>
<td>Stainless &amp; Alloy Steels</td>
<td>Kalling’s Reagent</td>
<td>Develops dendritic pattern. Darkens ferrite and martensite, austenite light, does not attack carbides.</td>
</tr>
<tr>
<td>Copper &amp; Copper Alloys</td>
<td>Acid Ferric Chloride</td>
<td>General etch for copper, brasses and bronzes.</td>
</tr>
</tbody>
</table>