University of Saskatchewan
Department of Mechanical Engineering
Standard Operating Procedure # MAT0005

Procedure Title: Submersion Etching Metallographic Specimens for Examination

Minimum Review Requirements:	**Annually**
Creation Date:	**January, 2011**
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1. Version History

Version #: .1
Supersedes: n/a

Handwritten amendments to the official procedures can be made by a single line through the text, along with the date, and initialed by the authorized individual making the correction. Changes are to be noted below. Formal changes to this SOP are made on the date of revision or sooner, where required.

Section	Changes made to official copy	Date	Initials

2. Introduction

This SOP provides a general method to chemically etch a metallurgical sample via submersion in a chemical etchant.

As different etchants have different toxicities and properties this document does not attempt to identify specific hazards, for such information the MSDS of the chemicals being used should be consulted.

This document is strictly to provide the method for submersion etching, including the collection of waste products and the cleaning of any labware used. It also addresses the removal of chemical waste and cleaning of equipment, as well as how to act should a chemical spill occur.

This SOP applies to all parties conducting this type of work, failure to follow this procedure or a disregard for laboratory rules and/or safety will result in loss of lab privileges.

All chemical etching **MUST** be conducted inside a fume hood and proper PPE must be worn at all times.

This procedure does not pertain to Hydrofluoric Acid (HF) containing etchants!

3. Definition

SOP:	Standard Operating Procedure
MSDS:	Material Safety Data Sheet
WHMIS:	Workplace Hazardous Materials Information System
PPE:	Personal Protective Equipment
WSEP:	Workplace Safety and Environmental Protection

4. Personnel

Persons authorized to perform this SOP:

By signing this form I acknowledge that I have read and understand this SOP, as well as the applicable MSDS's and that I will conduct myself in accordance with this SOP and the general laboratory rules.

NOTE: ALL SIGNATURES MUST BE PRESENT ON THE SOP LOCATED IN THE YELLOW BINDER IN ROOM 2C26 – Mechanical Engineering Materials Lab, digital copies of SOP's are made available for convenience only. Printed SOP's are valid for 24 hours only, after that time their accuracy must be verified with the **OFFICIAL HARDCOPY VERSION**.

Name (Print)	NSID	Dep't	Signature	Date
Robert Peace	Staff	ME		
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5. Safety

Many of the chemicals used in etching materials pose acute and chronic health hazards and proper protocol must be followed to **avoid** the following:

1. Contact with the skin and eyes
2. Inhalation
3. Ingestion
4. Contamination of work area – poses a health risk to others.
5. Some chemicals are not compatible with some materials (we typically use Nitrile gloves; however for some chemicals a different glove type **MUST** be used).
6. Glove compatibility charts are available from glove manufacturers and **MUST** be consulted prior to chemical usage.
7. See the relevant MSDS for the toxicological properties of the chemicals being used.

6. Procedure

Stage 1: Setting Up

1. Inspect PPE and verify all required materials are available.
2. Check emergency shower and eyewash station to ensure they have been inspected recently. If in doubt use a bucket to test the shower, **Do NOT use the shower without a catch bucket unless it is an emergency.**
3. Verify that Fume Hood is **ON** and operational.
4. Label the waste disposal container(s).
5. Review what to do in case of an accidental spill, both inside the fume hood and outside the fume hood.
6. Put on PPE.
7. Place Hazmat pads inside the plastic spill tray to use as a work surface.
8. Move materials into the fume hood.
9. Fill the primary and secondary rinse containers with water.

Stage 2: Procedure

10. Pour the reagent into the etching container, for all materials except Hydrofluoric Acid (HF) [which requires a separate SOP] a GLASS dish with a spout as shown in Figure 1 **Must** be used, it is NOT permissible to use a Petri dish or other round container without a spout.

Figure 1: Proper Etching Container – glass with spout.

11. Figure 2 shows the proper arrangement of labware with all components labeled, deviation from this arrangement is via special permission only.

Figure 2: Proper Etching Labware Arrangement

12. Using tongs pick up the specimen and dip it into the etchant, if etching will take under 5 seconds continue to hold onto the specimen, if it will take longer you can leave the specimen in the etchant and remove the tongs, the tongs can be placed in the large rinse container but NOT on the bench top (inside or outside of the fume hood).
13. Remove the specimen from the etchant and;
    a. dip into the primary rinse, swirling slowly (do not splash the rinse).
    b. Or if flowing water is required: Using a wash bottle with water in it gently spray the specimen into the large primary rinse container so that all waste is collected into the container, the specimen should be held with tongs for this procedure and care should be taken to not splash or mist while rinsing.
        i. Rinse for a minimum of 20 seconds (with a minimum of two grip locations) to ensure the etchant has been totally removed from the specimen
14. If using submersion rinsing: Place the specimen into the secondary rinse and swirl.
15. Complete a final rinse with 95% or 99% ethanol and blow dry the sample.

Stage 3: Clean-up

16. Figure 3 shows the proper arrangement of the waste collection container with appropriate funnel, if a 10L container is too tall to safely pour waste into while it is in the fume hood, a shorter waste container **MUST** be used.
    a. It is not permissible to locate the waste container outside of the fume hood, or to raise the fume hood sash above the proper working level to access the waste disposal container.
    b. For further information see Mat0001 for proper waste collection and disposal information.

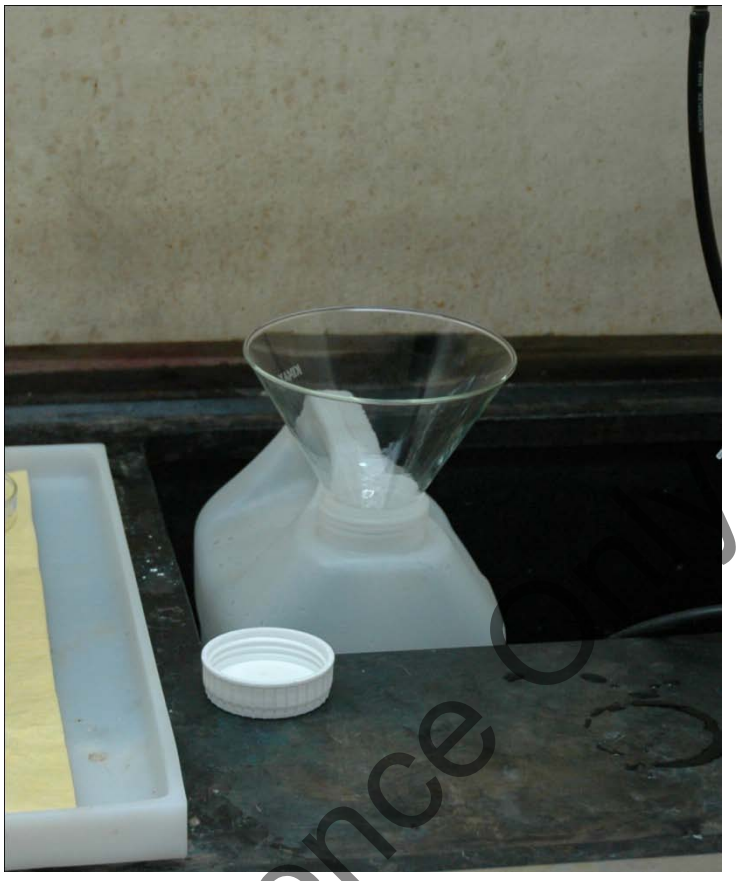

Figure 3: Proper Waste Container and Funnel Arrangement

17. Triple rinse all used beakers, funnels and utensils with tap water (while still in the fumehood) (or a squeeze bottle of de-ionized water if more convenient), collecting the rinse water in the labeled waste container. After triple rinsing, remove the glassware from the fume hood and air dry on paper towel on a bench top.
18. Put cap on waste disposal container and place in appropriate secondary containment inside the laboratory.
19. Any solid contaminated waste (gloves, pads, paper etc.) must be placed in a labeled **SEALED** plastic bag, sealed and placed in the secondary containment tub in the laboratory.
20. If you are unsure if a small spill has occurred wipe it up with a hazmat pad and package the waste as above, then re-wipe the area 3 times, collecting the paper towel/hazmat pads used.
21. In addition any putty, spatulas, or other disposable items that came into contact with a chemical must be placed in a bag labeled with the contaminant.

22. Any scales, power supplies, electrodes etc. must be triple rinsed/wiped and removed from the fume hood once etching is complete.
23. **Chemical Waste or Contaminated Articles Can NOT be disposed of down the drain or in a garbage bin.**
24. Remove all labels and signs from the work area.

7. Equipment or Materials Required

Prior to beginning any etching procedure the following equipment must be confirmed to be available.

The following Personal Protective Equipment (PPE) is required for this procedure:

- Fume Hood
- Lab Coat
- Closed Toed Shoes
- Long Pants
- Safety glasses (with splash protection), goggles or Face Shield
- This SOP
- MSDS Information – procedure dependent
- Operational Safety Shower
- Operational Eye Wash Station
- Proper Gloves – procedure dependent
- **NOTE: NONE OF THE GLOVES USED IN THE LABORATORY ARE INTENDED FOR SUBMERSION USE, THEY ARE FOR SPLASH/SPILL PROTECTION ONLY.**

This procedure also requires the following:

- Liquid Waste Disposal Container with appropriate label
- Solid Waste Disposal Container with appropriate label
- Etching specific glassware
- Etching specific rinse containers
- Sink for clean up
- Suitable tongs
- Glass Funnel for Waste Container

8. Highlights / Critical Control Points

NOTE: Gloves are worn for splash protection only, and MUST not be worn to touch equipment outside of the fume hood sink controls and what is necessary for etching. The microscope, door handles etc. must not be touched with gloved hands.

NOTE: If a glove has any liquid or powder substance on it (even water), it must IMMEDIATELY be removed and disposed of (either in a labeled solid waste container or in the garbage, depending on what is on the glove). If the substance on the glove is unknown it must be assumed to be a combination of all substances being worked with and disposed of in a labeled solid waste container.

NOTE: When mixing an acid or base into water or alcohol always add the acid/base to the water/alcohol and not the reverse.

9. References

University of Saskatchewan DHSE Documents:
Chemical Safety Code (Draft):
http://www.usask.ca/dhse/chemicalsafety/codes.php

Hazardous Waste Disposal:
http://www.usask.ca/dhse/file_view/download.php/Hazardous+Waste+Disposal+Manual+Aug+2007.pdf?id=3&view=1

Laboratory Safety Manual:
http://www.usask.ca/dhse/file_view/download.php/Laboratory_Safety_Manual.pdf?id=32&view=1