

Integrated Work Document (IWD) Part 1, Activity Specific Information

IWD #: 8-3-1698-C135H-1	Revision #: 2	Activity/Task Title: Metallography
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Work Document #			Planner/Preparer (Name/Z #/Date)		
			Ben Morrow 247959 01/09/2019		
TA 03	Building 1698	Room B122	Other Location(s) (TA) as required		

Activity Description/Overview:

Samples are prepared for metallographic analysis through a series of preparations steps that may include a combination of any of the following: cutting/sectioning, mounting, grinding, polishing, etching, and/or electropolishing. There are moderate hazards associated with some of these preparation steps (chemical, thermal, mechanical) and with some of the materials being prepared (chemical/toxicity).

Hazard Analysis (HA) Method Used: ☒ Brainstorming ☒ Other: Primary Hazard screen controls

List Names of HA Team (Attach sheet if necessary): Roberta Beal, Veronica Livescu, Ben Morrow

Date HA Performed: 12/11/2018

Responsible Line Manager (RLM) approval indicates confidence that the IWD/WCD was properly prepared; the hazard grading determination is appropriate; all activity task and co-located hazards have been identified and appropriately controlled; the work will be performed by an assigned Person in Charge (PIC) in accordance with the IWD/WCD, within ESH/S&S requirements, and within facility requirements and capabilities; and workers are competent. The RLM acknowledges the completion of a peer review (R&D only).

RLM or RLM Designee/Representatives (Signature/Z#/Date) Required: [Signature] 187042 5/29/2019

The Facility Operations Director (FOD) approval indicates that the work is appropriate to be conducted in the facility; the work is bounded by the Documented Safety Analysis (DSA); the work, performed in accordance with the IWD/WCD, will meet applicable Laboratory environmental, safety, and security requirements, as well as DOE Orders and other applicable regulatory requirements; and area-specific hazards have been identified and controlled.

FODs or FOD Representatives (Signature/Z#/Date/TA) Required: [Signature] 180035 6/12/19 143

Subject Matter Exper(s) (SME[s]) Review (Signature/Z#/Date) If Required: [Signature] 121475 5/14/2019

[Signature] 317874 5/28/19 14

Work Area Information (e.g., entry requirements, work area hazards and controls). Work area information can be obtained from placards, FOD direction, or other documentation (e.g., procedures). Form 2101, *Integrated Work Document (IWD) Part 2, FOD Requirements and Approval for Entry and Area Hazards and Controls, Non-Tenant Activity Form*, or Form 2102, *IWD Part 2, FOD Requirements and Approval for Entry and Area Hazards and Controls, Tenant Activity Form*, are optional forms to document work area information when warranted. If Forms 2101 or 2102 are not used, the work area information must be documented in the following section of this IWD.

Work Area Information (If necessary, document site hazards, controls, and/or entry requirements under this section)

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Hazard Determination by Hazard Grading Table: <input type="checkbox"/> Low-Hazard <input checked="" type="checkbox"/> Moderate-Hazard <input type="checkbox"/> High-hazard/Complex IWD Type: <input type="checkbox"/> Standing IWD <input checked="" type="checkbox"/> Standard IWD	Next Review Date: <u>5/20/2022</u> RLM or Designee and FOD or FOD Representative reapproval is required. Annual Review Completed (RLM or Designee Initial/Date): _____ Name of Person in Charge (PIC) (Print): <u>_Roberta Beal</u> Name of Alternate PIC : <u>Rod McCabe</u> Name of Alternate PIC : <u>Ben Morrow</u>	Classification Review Completed, if required. Reviewer Signature/Z#/Date _____
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Work Tasks/Steps Identify work steps/tasks in sequence when such sequencing contributes to safety, security, and/or environmental protection.	Hazards, Concerns, and Potential Accidents/Incidents Identify both activity and work- area hazards for each task/step.	Controls, Preventive Measures, and Bounding Conditions Specify preventive measures, controls for each hazard (e.g., lockout/tagout points, specific Personal Protective Equipment [PPE], Tamper Indicating Devices [TIDs], alarms, safes, recycling, waste minimization).	Reference Documents List permits, operating manuals, security plans, and other reference procedures.	Training List training and qualification requirements. (P300 Integrated Work Management, Section 6.1)
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Task 1: General Access, Laboratory Use, Emergency Response, and Waste Generation

1.1 General Lab Use	<ul style="list-style-type: none"> • Eye hazard (chemical splash, projectiles) • Chemical exposure due to inadvertent contamination or splash hazard • Inactive facility authorization basis • Ensuring known chemical composition and owner 	<p>When in active portion of chem lab, wear safety glasses with side shields, lab coat, long pants, and shoes that provide full foot coverage. Obey all postings.</p> <p>Low hazard activities (e.g. cutting, mounting, grinding, cleaning, and microscopy) are permitted without a second person present. Best practice is to inform another person that you're using the lab. A cognizant PIC can open the lab for use for moderate hazard activities. When no PIC is available, arrangements may be made to allow for low hazard work only with approval of PIC or RLM.</p> <p>All unattended chemicals and materials must have a legible label that is in English and includes: Contents (name of material) or a notebook reference if the contents have yet to be characterized as long as the notebook is readily available when the worker is not present, owner name, phone number, applicable hazards, if any (e.g. "contains lead", "carcinogen").</p>	P101-14 Chemical Management P101-6 Personal Protective Equipment P101-16 Local Exhaust Ventilation and HEPA Filtration Systems P409 Waste Management Optional training:	2810 Hazardous Waste Generator Training Plan MST-8 Metallography Facility Safety Orientation with Operation PIC
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	<ul style="list-style-type: none"> Unknown hazards associated with unlabeled or poorly labeled samples 	<p>If the sample is high consequence, the following additional information should be included on the label: Organizational affiliation, and contact information for owner.</p> <p>If samples are too small or numerous to be labeled individually, a secondary container may be used. The secondary container must safely and securely hold the sample(s) and the samples must be chemically compatible. The label on the secondary container should contain the labeling requirements listed above.</p>	<p>11108 MST PPE User Training Plan</p> <p>10341 Ventilation: Fume Hoods Training Plan Solvents, TP 2485 (274)</p> <p>SDS for all chemicals are located on the computer in the microscope room</p>	
1.2 Chemical Spills	<ul style="list-style-type: none"> Incidental spills of a small amount of material Greater than incidental spills 	<p>Incidental spills (an example of incidental would be a few drops from transferring a liquid between beakers) can be cleaned up by the involved workers.</p> <p>In the event of a spill of corrosive or hazardous materials that is greater than incidental, the following procedures should be followed (assuming no contact with eyes or skin):</p> <ol style="list-style-type: none"> 1) Evacuate the lab of all personnel, stay uphill and upwind 2) Assure that the lab entrances are secured, 3) Hit the red 'Emergency Purge' button near the west entrance if evacuating in that direction, and 4) Immediately notify the PIC and RLM of the spill. Also notify IH, FOD on call (664-3865), and STO Duty officer at 664-4444, and EM&R (7-6211) may be notified at this point, depending on the severity of the spill. <p>Only PICs should handle greater than incidental spills with proper training, appropriate PPE, proper spill containment that does not contain radioactive material or category 1 chemicals or spill into the hallway.</p> <p>Tenants are not allowed to control spills that involve category 1 chemicals. In the event of a category 1 spill, in addition to steps above, also call 911.</p> <p>Acid and solvent spill kits are located above the Flammable Storage cabinet and should only be used by one of the PICs, and only for</p>	<p>Hazardous Materials Spill Response Emergency Action Procedure Course #54754</p> <p>STO Division Building Emergency Plan for STO Complex, STO-BEP-001</p>	<p>All trainings required from Task 1.1</p>

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	<ul style="list-style-type: none"> Solutions containing nanoparticles 	<p>simple spills that the PICs feel comfortable addressing. All complex spills will be handled by EM&R.</p> <p>In the event of a spill of solution containing nano particles, notify a PIC to clean the spill. All waste in contact with nanomaterials as a result of a spill must be disposed of as hazardous waste (e.g. gloves, disposable lab coats, swabs, Kimwipes, blotter paper, beakers, flasks, tape, sample tips, sample holders, etc.). The solid material must be kept in a sealable container with a LANL UNP label and stored in the SAA for pick-up.</p>		
1.4 Emergencies		<p>A safety shower is located near perchloric hood in C135H. Eyewashes are located at the two sinks nearest the west entrance.</p> <p>In the event that you are exposed to hazardous chemicals:</p> <p>1) If a chemical is splashed in your eye, irrigate for 20 minutes, holding the eye open. Don't wait for pain or other noticeable effects. While irrigating, have someone else contact 911 and notify the RLM and the PIC.</p> <p>2) For other significant skin contact resulting in the potential for chemical burns, remove any affected clothing and remain under the shower until emergency personnel arrive (shower a minimum of 15 minutes, or 30 - 60 minutes for severe burns). While showering, have someone else contact 911 and notify the RLM and PIC.</p> <p>2a) HF burns can be fatal, must see Occupational Medicine ASAP.</p> <p>For HF skin contact, rinse affected area, apply calcium gluconate gel to affected area to neutralize HF exposure, and activate the EMS system. Serious exposures may initially appear insignificant. Calcium gluconate gel, along with posted instructions for its use, are located above the flammable cabinet with the acid spill kit.</p> <p>3) If incident is not severe, the affected personnel should be taken directly to Occupational Medicine after the above steps. Notify the RLM and PIC, and the RLM should accompany the affected individual to Occ. Med.</p>		

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1.5 Waste Disposal Common wastes: a) Polishing waste b) Solvents and chemicals c) Mixtures (i.e. etchants)	Improper disposal can lead to spilling of hazardous and high volumes of hazardous waste or dangerous situations	Liquid non-hazardous waste from mechanical sample preparation operations can be disposed of down the RLW lines in the room according to existing waste profiles; Solid non-hazardous waste can be discarded in the general trash bins. This can be performed by all users. Only the PICs are allowed to dispose of chemical and solvent waste; students and postdocs can dispose of chemical waste with direct supervision by PIC.		
Task 2 Chemical polishing and etching				
2.1 Chemical use (including solvents, acids, bases, solutions and mixtures, other chemicals) Procedure for etching: -Prepare work area - Don appropriate PPE -Swab with or immerse sample in etchant -Rinse with water or solvent, as appropriate -Dry sample -Clean work area and store chemicals	<ul style="list-style-type: none"> • Skin absorption of chemicals by wearing no gloves or improper gloves for the chemical at hand. • Breathing vapors/dust from chemicals by not working in a properly ventilated area • Eye hazard (chemical splash, projectiles) • Chemical exposure due to inadvertent contamination or splash hazard • Fire hazard from flammable chemicals 	Workers should consult the SDS of any unfamiliar chemicals for specific hazards and considerations. Appropriate PPE should be worn when handling chemicals. This includes lab coat, safety glasses, and appropriate gloves (reference glove chart). Chemical splash goggles and a face shield should be worn when pouring/mixing chemicals. Operations that could produce fumes (including mixing chemicals, etching) should be performed in a chemical fume hood. Check to see if the hood is operating properly. Use the hood sash in the range listed on the certification notice. Secondary containment must be used when working with chemicals. Keep flammable solvents away from heat sources. Store chemicals in their appropriate storage areas/cabinets when not in use	SDS located on computer in B122A. LANL SDS database: http://hq.msdsonline.com/losalamosnational/absl/Search/Default.aspx Metallography Handbook : Facts about Flammable and Combustible Liquids	All trainings from Task 1 4261 Chemical Workers, Authorized (Haz Com) Training Plan
2.2 Nanoparticles	<ul style="list-style-type: none"> • Colloidal silica and alumina polishing 	Use colloidal polishing suspensions in a wet state and use wet cleanup methods on any dried suspension to prevent airborne	P101-29, Working with Nanotechnology	Engineered Nanoparticle Worker TP 10846 (5410)

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	suspensions contain engineered nanoparticles.	dispersion of engineered nanoparticles. Everything that comes in contact with nanoparticles MUST be cleaned thoroughly and not be allowed to dry. All solutions containing nanoparticles will be identified with a LANL UNP label.	Materials and Processes	
2.3 HF Acid Use	<ul style="list-style-type: none"> HF liquid or vapors may burn the eyes or skin. (HF) is a particularly dangerous acid because of its unique ability among acids to penetrate tissue. This ability to penetrate tissue allows HF to cause severe systematic toxicity from even relatively small dermal exposures. If skin penetration is sufficiently deep, decalcification of the bones may result. Corrosive to the nose, throat, and lungs. HF attacks glass, concrete, and many metals (especially cast iron). This can cause a spill hazard 	<p>Don appropriate PPE. In case of chemical contact with HF, rinse affected area (limit to 5 minutes if calcium gluconate gel is available), apply calcium gluconate gel (on the wall directly behind both hoods), and activate emergency response. Calcium gluconate gel is inspected every 6 months and replaced every year.</p> <p>Additional PPE, such as coveralls, gauntlets, and boots, face shield in conjunction with splash goggles for open processes or open hood sash. Extended use of concentrated acid >70%-Neoprene gloves. Hydrofluoric acid 30-70% - use Butyl rubber, Neoprene, or Viton/butyl gloves should be considered according to conditions.</p> <p>Use containers like platinum, wax, polyethylene (PE or HDPE), polypropylene (PP), polymethylpentane, and Teflon, which will resist the corrosive action of HF. Never use glass. Sodium bicarbonate should never be used with an HF spill since it does not bind the fluoride ion and can generate toxic aerosols.</p>	<p>Hydrofluoric Acid Safety: https://int.lanl.gov/training/its/flyers/HF_Acid_IF_028_11-07.R0.pdf</p> <p>P101-14 Chemical Management</p>	Curriculum 44498 First Aid Response to Hydrofluoric Acid
2.4 Perchloric Acid Use	<ul style="list-style-type: none"> Under extreme conditions (i.e. boiling at 397°C) not covered under this IWD, there is a small chance for formation of perchlorate crystals. Dilute perchloric/acetic acid mixtures may freeze over time at refrigerator temperatures. This can result in the formation of highly concentrated residual liquid. 	<p>Don proper PPE. Perchloric acid work can only be performed in designated Perchloric Fume Hoods. Hood #17020 in room C135H is designated for this type of work. No organic chemical processes, separate from those involved in the perchloric acid processes, should occur in this hood. Ensure the hood is operating correctly according to the inspection sticker. Perchloric hoods must be washed down after use per posted procedure. Drains should be checked to make sure water can drain freely prior to wash down.</p> <p>Normal usage conditions are at or below room temperature and stock perchloric acid and solutions containing perchloric acid are below the perchloric content necessary for explosive regime.</p>		

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		Perchloric and perchloric mixtures are stored in the refrigerator. The refrigerator is kept at a temperature higher than the freezing point of the perchloric/acetic mixture.		
2.5 Mixing Etchants General Procedure: <ul style="list-style-type: none">• Prepare work area• Don appropriate PPE• Mix in the hood• Mix the absolute minimal amount<ul style="list-style-type: none">-Add stronger acids/bases to weaker/solvent; Never add solvents to acid/base-For exothermic reactions during mixture, use ice bath to cool solution• Ensure appropriate label is affixed to solution and store as appropriate	<ul style="list-style-type: none">• Exploding of mixed chemicals resulting in acid burns, eye injury, cuts from broken glassware.• Many metal etchants are highly reactive mixtures and can become unstable over time.	<p>Only those directly approved by the PIC are allowed to mix or use etchants without supervision. As a moderate risk activity, operation must only be performed during normal working hours, in case of an accident.</p> <p>Don proper PPE. In addition to the basic PPE required for chemical use, users should consider the following extra precautions: Chemical splash goggles and faceshield, and thick rubber or nitrile gloves (per glove selection chart and SDS)</p> <p>Only etchants approved by PICs may be mixed. Etchant preparation is controlled through a labeling system maintained by the PICs. Etchants should be stored in a bottle with a standardized label provided by a PIC. The label contains information on contents, owner, preparation date, and expiration date. In addition, the label may include usage, storage, and/or disposal information.</p> <p>Mix chemicals slowly with stirring so they do not become unstable. If mixing is strongly exothermic, mix in beaker placed in an ice bath. Add acids/bases to solvents; do not add solvents to acids/bases. In general, add stronger acid/base to weaker. Use a clean pipette and graduated cylinder for each new chemical being mixed.</p> <p>Users must be aware of the mixing hazard and disposal path of an etchant prior to creating it.</p> <p>Store properly (e.g., flammables in the flammable cabinet away from oxidizers; acids separate from bases). Mixed etchants are stored in the explosion proof refrigerator located behind the consumables cabinet.</p> <p>Use the ventilation fume hood for mixing chemicals, and keep the hood closed to the extent possible to minimize the splash hazard.</p>	<p>Special Material Use Without Supervision Document</p> <p>LANL Chemical Management Tools: https://int.lanl.gov/safety/industrial_hygiene_and_safety/chemical-safety/chemical-management-tools.shtml</p> <p>LANL Chemical Compatibility Tools: https://int.lanl.gov/safety/industrial_hygiene_and_safety/chemical-compatibility-tools.shtml</p> <p>Metallography Lab Handbook: Working with Chemicals</p> <p>EPA Chemical Compatibility Chart located on the cabinet under the hood & next to white board.</p> <p>Approved Etchants List.</p>	<p>All trainings from Task 1</p> <p>4261 Chemical Workers, Authorized (Haz Com) Training Plan</p>

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Task 3: Electropolishing				
<p>Three options for electropolishing:</p> <ul style="list-style-type: none"> a) live leads b) jet polisher c) bulk polisher <p>General procedure:</p> <ul style="list-style-type: none"> • Prepare work area • Don appropriate PPE • Add electrolyte to appropriate location of polishing equipment -Cool to desired temperature, if necessary • Insert sample • Apply voltage for desired time. -Follow manufacturer instructions for commercial equipment • Remove sample and rinse with water or solvent, dry sample. • Clean equipment after use 	<p>Outages or malfunction might produce abnormal operating conditions and introduce new hazards.</p> <p>Electrical: exposed probes could lead to possible exposure to electric shock</p> <ul style="list-style-type: none"> • Potential cryogen burn when using cryogenics to chill solutions • Suffocation hazard exists for cryogenics • Chemical exposure from electrolyte 	<p>As a moderate risk activity, operation must only be performed during normal working hours, with a PIC on the premises, in case of an accident.</p> <p>The MST-8 ESO has approved all equipment. Conductors are shielded to the maximum extent possible.</p> <p>Electro-polishing at under 100V DC is allowed for non-energized electrical workers. Equipment is rated to a maximum of 1.7A which is less than 1kW and equivalent to work under hazard class 2.1b (non-energized) per P101-13. For equipment use outside these conditions the following requirements are in affect: energized electrical worker training and consultation with PICs before work is performed.</p> <p>Wear appropriate PPE, including safety glasses, face shield, long sleeves, and cryogen-appropriate gloves when transferring liquid nitrogen from dewar or handling cryogenics.</p> <p>The Preplab does not provide liquid nitrogen. To obtain cryogen for use in the met lab, the users must be authorized on separate IWD (see references).</p> <p>Use minimum necessary quantities of cryogen. If possible, use in a fume hood to ensure vaporized nitrogen is pulled from the room.</p> <p>Perform chemical operation in accordance with Task 2. Clean equipment thoroughly after use to avoid contamination.</p>	<p>P101-13 Electrical Safety Program</p> <p>P101-5 Cryogenics</p> <p>IWD# 10-3-3234-DOCK-1, Inert Cryogen/Compressed Gas Cylinder Handling Operations</p>	<p>All trainings from Task 1</p> <p>2899 R&D Electrical Workers: Non-Energized Training Plan</p> <p>2720 Cryogenic Fluids Worker Training Plan</p>

Task 4: Equipment Maintenance/Modification				
	<ul style="list-style-type: none"> • Potential electrical hazard from energized equipment • Potential pressure hazard from air hookups 	<p>Only those directly approved by the PIC are authorized to make modifications to or maintain any equipment or system in the metlab without supervision. Systems should be discharged appropriately by authorized workers (e.g. qualified energized electrical, pressure</p>		<p>All trainings from Task 1</p> <p>2306 Low Pressure Systems Training Plan</p>

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		system worker) to verify that they are in a safe configuration before work begins. Troubleshooting of energized electrical systems must be performed by R&D Electric: Energized Worker. The pressure systems are designed and maintained according to LANL policy P101-34 and Engineering Standards Manual Chapter 17.		2899 R&D Electrical Workers: Non-Energized Training Plan



Integrated Work Document (IWD) Part 3, Validation and Work Release

IWD #MST-8-3-1698-C135H-1

Revision #:2 Work Release

By signing below, I verify this activity is compatible with current facility configuration and operating conditions.

FOD designated Ops Mgr or other facility point-of-contact for work area

Signature/Z#/Date (If required by FOD): _____

Note: For Standing IWD, release may be given concurrently with signatures on Part 2.

By signing below, I have verified the following:

- I have verified authorization by ensuring approval signatures of the RLM and FOD.
- I have jointly conducted a validation walkdown with workers to confirm the IWD can be performed as written, required initial conditions and other prerequisites are in-place.
- The assigned workers are authorized and are qualified to perform the work in a safe, secure, and environmentally responsible manner.
- I have conducted the pre-job briefing, and all workers (including support workers) have been briefed.
- I have ensured coordination with any required FOD work area representatives (e.g., area work coordinators).

Primary PIC (Signature/Z#/Date) Required: [Signature] 178257 6/13/19Alternate PIC Signatures **acknowledges** PIC authority is assumed for the first time (Note: Alternate PICs are required to sign only once, but formal handoff includes conferring with previous PIC to obtain all required information associated with the handoff).Alternate PIC (Signature/Z#/Date) Required: [Signature] 247959 6/13/2019Alternate PIC (Signature/Z#/Date) Required: [Signature] 322242 / 6-13-19**Pre-Job Brief Content**

- What are the critical steps or phases of this activity?
- How can we make a mistake at that point?
- What is the worst thing that can go wrong?
- What controls, preventive measures, and bounding conditions are needed?
- What work permits are required and how will we meet their requirements?
- What are the handoffs and coordination requirements among workers and multiple PICs?
- Are there hold-points including those that require sign-offs?
- What are the pause/stop work responsibilities and expectations (e.g. for unanticipated conditions or hazards)?
- How would we respond to alarms and emergencies?
- Are there lessons learned from previous similar work?
- Is other information needed to perform this activity in a safe, secure, and environmentally responsible manner?
- Does everyone agree to the work tasks/steps, hazards, and controls and commit to follow them?

Pre-Job Brief Attendance RosterBy signing below **as required**, I agree to the following:

- I agree to follow the work steps and implement the controls as written as applicable to my work assignments.
- I agree to pause/stop work when conditions or hazards change or when I encounter unexpected conditions during the execution of work, or when work cannot be performed as written, or instructions become unclear during execution.
- I confirm that I am authorized, qualified, and fit to perform the work.

Worker (Signature/Z#/Date) [Signature] 178257 6/13/19

Worker (Signature/Z#/Date)

Worker (Signature/Z#/Date) [Signature] 322242 / 6-13-19

Worker (Signature/Z#/Date)

Worker (Signature/Z#/Date)

Worker (Signature/Z#/Date)

Worker (Signature/Z#/Date)

Worker (Signature/Z#/Date)