INEEL--97054490

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OSTI BENEFITS CHECKLIST

CRADA NO.: 95-CR-01

PARTNER: Johnson Controls

PRINCIPAL INVESTIGATOR: Kevin McHugh

ACCOUNT EXECUTIVE: Carolyn Roberts

MARK "X" in the appropriate response.

BENEFITS REALIZED? ___ YES ___ NO

LIST: Experience gained in handling/spray forming lead alloys.

CONSTRAINTS/EXTERNAL INFLUENCES:

___	Technical or manufacturing problems	___	Changes in market conditions
___	Funding Availability	___	Competing Technology
___	Personnel Changes	___	Legislative/Regulatory Impacts
___	Work Scope Changes	___	Changes in Partner Objectives
___	Other:		

FOLLOW-ON ACTIVITIES

___	CRADAS	#___	___	Licenses	#___
___	Cost-shared Contracts	#___	___	Copyrights	#___
___	Invention Disclosures	#___	___	Reimbursable WFO	#___
___	Technical Assistance	#___	___	Use of Facilities	#___
___	Patent Applications	#___	Non US #___		
___	Other:				

Were Any Awards Given? ___ YES _X_ NO

List:

MASTER

Completion Date: 4/10/96

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Spray Forming Lead Strip
Final Report

CRADA 95-CR-01

Experimental

A cooperative research project was conducted between the Idaho National Engineering Laboratory (INEL) and Johnson Controls, Inc. (JCI) to adapt the INEL spray forming process to produce near-net-shape lead alloy strip. The emphasis of the work was to spray form lead strip samples at INEL, using a variety of spray conditions, for characterization at JCI.

An existing glove box apparatus was modified at INEL to spray form lead. The main spray forming components were housed inside the glove box. They included a spray nozzle, tundish (crucible), substrate assembly, gas heater and furnaces to heat the nozzle and tundish. To spray form metal strip, liquid metal was pressure-fed at a controlled rate through a series of circular orifices that span the width of the nozzle. There the metal contacted high velocity, high temperature inert gas (nitrogen) which atomized the molten material into fine droplets, entrained the droplets in a directed flow, and deposited them onto glass plates that were swept through the spray plume to form strip samples. In-flight convection cooling of the droplets followed by conduction and convection cooling at the substrate resulted in rapid solidification of the deposit. During operation, the inside of the glove box was purged with an inert gas to limit the effects of in-flight oxidation of the particles and spray-formed strips, as well as to protect personnel from exposure to airborne lead particulate. Remote controls were used to start/stop the spray and control the speed and position of the substrate. In addition, substrate samples were loaded into the substrate translator manually using the gloved side ports of the box. In this way, the glove box remained closed during a series of spray trials, and was opened only when loading the crucible with a lead charge or when removing lead strip samples for shipment to JCI.

A bench-scale (0.66" wide) linear de Laval (converging/diverging) nozzle and crucible assembly were designed and constructed in-house from high density graphite. The substrate assembly and drive, also designed and built at INEL, had been used previously to spray form strips of low melting point metals. About 10 lbs. of a lead alloy were cut and loaded into the crucible that was then sealed to the nozzle using a graphite gasket. The crucible lid was also sealed to the crucible using a graphite gasket. Melt flow into the nozzle was started/stopped using a pneumatically driven Al_2O_3 stopper rod assembly; metal throughput was controlled by adjusting the magnitude of positive pressure applied to the liquid metal. Melt temperature, atomizing gas temperature, nozzle inlet pressure, crucible pressure, and nozzle-to-substrate distance were varied in an effort to optimize strip properties.

Initial spray trials were conducted with pure tin to verify satisfactory operation of the equipment. Seventeen spray runs were conducted over three days. A wide variety of experimental conditions were explored; they are summarized in Table 1. For all runs, room temperature glass plates were used for substrates. Spray formed tin strips were typically about 13" long, and varied in width

Table 1. Tin strip- spray parameters and properties.

INEL ID #	Melt Temp. (°C)	Nozzle Temp. (°C)	Nozzle Gas Temp (°C)	Nozzle Inlet Press. (psia)	Tundish Press. (psia)	Nozzle-to-Substrate Distance. (in.)	Strip Thickness (in.)
1J1225A	411	416	416	20	12.2	13	0.018
1J1225B	418	411	411	20	12.2	20	0.020
1J1225C	400	412	412	20	13	20	0.035
1J1225D	403	414	414	20	13	20	0.045
1J1225E	421	404	404	20	13	10	0.025
1J1375A	401	295	397	25	12.5	13	0.012
1J1375B	404	292	395	25	12.5	20	0.017
1J1375C	404	295	395	25	13	20	0.031
1J1375D	430	300	412	25	13	20	0.041
1J1375E	402	321	400	25	13	10	0.022
1J1425A	435	440	429	22.5	13	20	0.025
1J1425B	427	383	427	22.5	12.5	20	0.020
1J1425C	428	387	428	22.5	12.5	13	0.025
1J1425F	433	430	428	18	12.5	20	0.026
1J1425G	434	406	428	22.5	12.5	20	0.017
1J1425H	431	390	436	22.5	12.5	20	0.020
1J1425I	432	398	433	22.5	12.5	20	0.045

over the range 3" to 5" depending on conditions. Strip thickness, which ranged from 0.012" to 0.045", was most easily controlled by varying the rate at which the plates were swept through the spray plume in conjunction with the throughput of metal. Droplet velocity and cooling rate were controlled by varying the nozzle pressure, while droplet temperature at impact reflected the interplay of melt temperature, nozzle pressure, metal throughput, and distance to the substrate.

Depending on conditions, as-deposited strip density ranged from 91 to 98% of theoretical density. Microstructural evaluation of polished samples indicated porosity was concentrated at the deposit/substrate interface in some samples while others, typically those produced at low deposition rates or large nozzle-to-substrate distances exhibited a more random distribution of pores. To reveal grain structure, the samples were etched using a 2 vol. % solution of HCl in methanol. A typical photomicrograph is shown in Figure 1a. The average grain size was 7μm and appeared uniform throughout the deposit. Under relatively "hot" spray conditions, a banded structure of fine and coarse grains was observed; Figure 1b is an example.

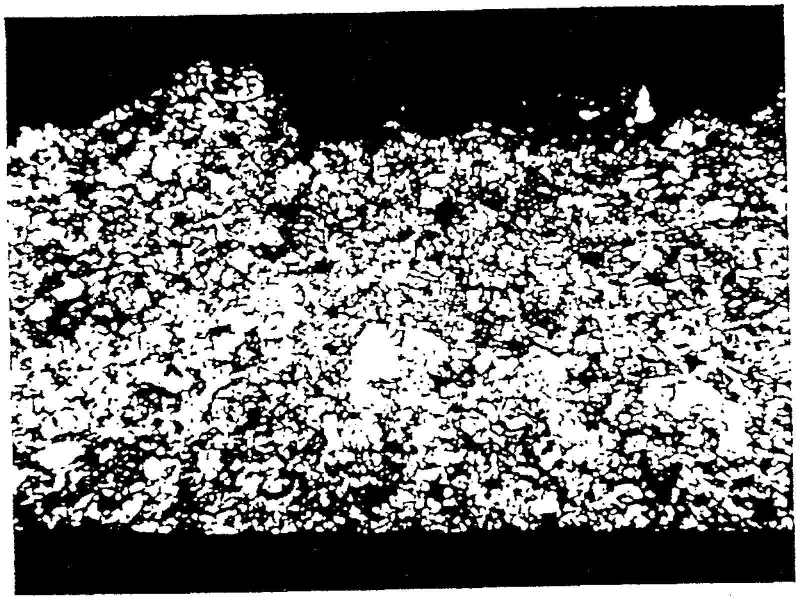
The spray runs with tin provided guidance for appropriate experimental conditions for lead alloy runs. Two lead alloys, supplied by JCI, were spray formed. Measured compositions of the alloys (by weight) were:

CAG alloy: Pb-0.04Ca-0.68Sn-0.01Al-0.03Ag
Pb1.4Se alloy: Pb-1.42Sb-0.15As-0.20Sn-0.02Se.

Goals included spray forming lead strip of about 0.030" to 0.060" thickness, with as-deposited density ≥98% of theoretical density, and with a homogeneous, fine grain structure. Post processing, such as rolling, was not desirable and was not conducted at INEL. In addition, production rates consistent with or greater than current continuous casting practices were desirable but were not the focus of this feasibility study.

In all, sixty-five spray runs with lead were conducted. All property evaluation of the spray-formed lead strips was conducted at JCI. These evaluations included strip microstructure, porosity, hardness, tensile properties, and corrosion resistance.

The initial spray runs with lead were conducted with both Pb1.4Se and CAG alloys. The alloys, supplied in the form of "piglets," were sectioned and loaded into the crucible. Ten runs were conducted with the Pb1.4Se alloy and eight with the CAG alloy. Table 2 summarizes experimental parameters and resultant strip thickness. Based on results using tin, a nozzle pressure of 20 psia was used for most of the runs. A melt superheat of about 100^{o}C was used and atomizing gas temperature was maintained above the liquidus temperature of the alloys. Two tundish pressures and six nozzle-to-substrate distances were explored, producing strips that ranged in thickness from 0.037" to 0.124". All runs were single-pass, i.e., the substrate was swept through the spray only once, except for one run with CAG alloy (1J2085B-8) in which the substrate was passed through the spray plume three times generating a strip 0.131" thick. All runs were conducted using room temperature window pane glass plates as substrates that were translated through the spray at the same rate. All strips were sent to JCI for analysis after measuring their thickness.



(a) Experiment 1J1375A. Uniform grain structure with 7μm average grain size. 200X.

(b) Sample 1J1225A. Nonuniform grain structure. Coarese grains are at the exposed deposit surface. 100X.

Figure 1. Photomicrographs of polished/etched tin strip samples.

Table 2. Experimental parameters from initial spray runs with lead alloys.

INEL ID#	Material Sprayed	Melt Temperature (°C)	Nozzle Temperature (°C)	Nozzle Gas Temperature (°C)	Nozzle Inlet Pressure (psia)	Tundish Pressure (psia)	Distance from Nozzle Exit to Substrate (in)	Strip Thickness (in)
1J2085A-1	Pb1.4Se	428	420	430	20	13	22	.037
J2085A-2	Pb1.4Se	436	424	436	20	13	20	.053
1J2085A-3	Pb1.4Se	430	426	429	20	13	15	.067
1J2085A-4	Pb1.4Se	414	419	419	20	13	18	.047
1J2085A-5	Pb1.4Se	422	391	429	20	13	12	.075
1J2085A-6	Pb1.4Se	418	388	428	20	13	9	.113
1J2085A-7	Pb1.4Se	428	400	430	20	16.5	12	.124
1J2085A-8A	Pb1.4Se	430	386	429	25	13	20	*
1J2085A-8B	Pb1.4Se	427	362	412	25	15	15	*
1J2085A-9	Pb1.4Se	425	372	425	25	15	12	.058
1J2085B-1	CAG	552	522	552	20	13	22	.054
1J2085B-2	CAG	570	519	558	20	13	20	.056
1J2085B-3	CAG	575	535	555	20	13	18	.033
1J2085B-4	CAG	571	541	563	20	13	15	.035
1J2085B-5	CAG	575	551	558	20	13	12	.042
1J2085B-6	CAG	574	538	554	20	13	9	.044
1J2085B-7	CAG	568	512	549	20	16.5	12	.037
1J2085B-8	CAG	550	480	535	20	16.5	12	.131

Notes: Speed Controller set at 25%. * Not Measured because strips were on opposite sides of glass plates.

During gas atomization, a liquid is disintegrated into relatively fine droplets by the action of aerodynamic forces that overcome surface tension forces that consolidate the liquid. The liquid's viscosity and density also influence atomization behavior but typically play a more secondary role. Viscosity affects both the degree of atomization and the spray pattern by influencing the amount of interfacial contact area between the liquid and gas. Highly viscous liquids oppose change in geometry more efficiently, making the generation of a uniform spray more difficult for a given set of flow conditions. Density influences how the liquid responds to momentum transfer from the gas. Light liquids accelerate more rapidly in the gas jet. Disintegration efficiency is reduced because atomization takes place at lower relative velocities.

Pb1.4Se proved to atomize more readily than CAG alloy, suggesting lower viscosity and surface tension. However, sprays with both alloys were coarser than were observed with tin. While the room temperature viscosity of pure lead is about 44% higher than that of tin, the kinematic viscosities are about the same. In addition, the surface tension of Sn is higher than that of Pb (560 and 458 mN/m, respectively). Thus, alloying additions to lead seemed to have a very dramatic effect on viscosity and surface tension of the melts. Under all spray conditions used in the initial runs, the CAG alloy sprays were very coarse, with large variation in droplet size in the spray plume. However, for all tin strips and nearly all lead strips, the deposits had a specular finish at the deposit/substrate interface suggesting a relatively low interfacial tension with good wetting.

A good predictor of break up tendency of liquids is given by the Weber number, "We," the ratio of inertial forces to surface tension forces:

$$We=\rho V^2 D/2\sigma$$

where ρ is the gas density, V is the initial relative velocity between the flow field and the drop, D is the diameter of the drop, and σ is the surface tension of the drop. This equation suggests that increasing the gas velocity by increasing the nozzle pressure while increasing the gas/metal mass flow ratio to reduce the drag effect should improve atomization efficiency of the metal. Other recommendations regarding future experiments, however, are difficult to make without knowing microstructural results of the lead strips and how these features correlate with experimental conditions.

In an effort to improve the as-deposited density, the second set of spray trials with lead were conducted using a higher tundish pressure, typically 17 psia, and shorter nozzle-to-substrate distances (6" to 12"). Nozzle inlet pressures were varied over the range 16.5 to 24 psia. The effect that heating the substrate had on strip microstructure, porosity, and other properties was evaluated. Substrates (glass plates) were heated prior to deposition in some experiments by positioning them 6" from the nozzle exit and allowing hot gas to impinge on their surface as they were raised and lowered through the hot gas jet. Spray parameters are summarized in Tables 3a and 3b for Pb1.4Se alloy and Table 4 for CAG alloy. A total of twenty-two Pb1.4Se alloy strips and eight CAG alloy strips were spray formed. All samples were boxed and shipped to JCI for evaluation.

Table 3a. Second set of Pb1.4Se alloy spray runs using glass substrates.

INEL ID #	Melt Temp. (^{o}C)	Nozzle Temp. (^{o}C)	Nozzle Gas Temp. (^{o}C)	Nozzle Inlet Press. (psia)	Tundish Press. (psia)	Nozzle-to-Substrate Distance (in.)	Speed Controller Setting (%)	Heated Substrate (Y or N)
1J2215A-1	428	409	430	20	17	6	100	Y
1J2215A-2	438	421	430	20	17	9	100	Y
1J2215A-3	428	377	433	20	17	9	25	N
1J2215A-4	430	413	425	20	17	9	50	N
1J2215A-5	428	405	433	20	17	6	100	N
1J2215A-6	432	378	429	20	17	6	50	N
1J2215A-7	425	425	432	16.5	17	9	100	N
1J2215A-8	432	393	430	16.5	17	9	100	Y
1J2215A-9	430	388	425	24	17	9	100	Y
1J2215A-10	431	406	430	24	17	9	100	N
1J2215A-11	426	427	431	20	20	12	50	N
1J2215A-12	480	431	470	20	17	6	100	Y
1J2215A-13	477	433	470	20	17	6	50	Y

Table 3b. Pb1.4Se alloy spray runs using glass substrates.

INEL ID #	Melt Temp. (°C)	Nozzle Temp. (°C)	Nozzle Gas Temp. (°C)	Nozzle Inlet Press. (psia)	Tundish Press. (psia)	Nozzle-to-Substrate Distance (in.)	Speed Controller Setting (%)	Heated Substrate (Y or N)
1J2215B-1	401	421	404	16.5	17	6	50	Y
1J2215B-2	403	N/A	415	16.5	17	12	50	N
1J2215B-3	402	N/A	412	24	17	6	50	Y
1J2215B-4	401	N/A	404	24	17	12	50	N
1J2215B-5	440	N/A	440	20	17	9	50	Y
1J2215B-6	442	N/A	447	20	17	9	50	N
1J2215B-7	478	N/A	490	16.5	17	6	50	N
1J2215B-8	480	N/A	483	16.5	17	12	50	Y
1J2215-10	482	N/A	482	24	17	12	50	Y

Table 4. Second set of CAG alloy spray runs using glass substrates.

INEL ID #	Melt Temp. (°C)	Nozzle Temp. (°C)	Nozzle Gas Temp. (°C)	Nozzle Inlet Press. (psia)	Tundish Press. (psia)	Nozzle-to-Substrate Distance (in.)	Speed Controller Setting (%)	Heated Substrate (Y or N)
1J2225A-1	404	392	406	16.5	17	6	50	Y
1J2225A-2	409	N/A	401	16.5	17	12	50	N
1J2225A-3	401	N/A	395	24	17	6	55	Y
1J2225A-4	401	N/A	404	24	17	12	50	N
1J2225A-5	437	N/A	444	20	17	9	50	Y
1J2225A-6	439	N/A	408	20	17	9	50	N
1J2225A-7	481	N/A	478	16.5	17	6	50	N
1J2225A-8	476	N/A	481	16.5	17	12	50	Y

Results indicated that most CAG and some Pb1.4Se lead alloy samples contained unacceptable levels of porosity while other Pb1.4Se samples appeared acceptable. Based on microstructural evaluation conducted at JCI, it was decided that subsequent runs would be conducted using Pb1.4Se alloy and, for the most part, conditions used during Run 1J2215B-5 (see Table 3b).

A final set of runs was conducted. Experimental parameters are summarized in Table 5. To examine the effect substrate material properties have on spray formed strip quality, a variety of substrate materials were used. They included polyimide, plexiglass, copper, stainless steel, and glass. In one set of experiments, the substrates were maintained at room temperature prior to deposition. In another set of experiments, stainless steel substrates were preheated to 50, 100, 150, 200, and 250°C using a Watlow ceramic fiber heater. All strip samples were sent to JCI for evaluation.

Safety Evaluations and Issues

As a result of the toxic nature of lead, extensive evaluations were conducted by both the Department of Energy (DOE) and Lockheed Martin regarding the impact spray forming lead has on the environment, and the safety and health risks posed to employees conducting the experiments. It was required that the proposed actions would not: 1) threaten a violation of applicable statutory, regulatory, or permit requirements for environmental, safety, and health, including requirements of DOE orders; 2) require siting and construction or major expansion of waste storage, disposal, recovery, or treatment facilities; 3) disturb hazardous substances, pollutants, contaminants, or CERCLA-excluded petroleum and natural gas products that persist in the environment such that there would be uncontrolled or unpermitted releases; 4) adversely affect environmentally sensitive resources. An internal Hazard Analysis (HA) was conducted which included an Environmental Checklist (EC). They indicated spray forming lead strip was potentially hazardous, thereby requiring a Safety Evaluation (SE) be conducted; results of the HA checklist, EC, SE, and other NEPA documentation are given in Appendix A.

During experiments, it was required that respirators equipped with HEPA filters, disposable gloves, and lab coats be worn by all personnel involved with the handling and spray forming of lead. The use of respirators required specialized training. It was also required that respiratory protection be worn when testing was conducted to measure exposures to lead. In addition to personnel safety, of particular concern was the possibility of particulate discharge to the atmosphere. Completion of the EC dictated that experiments should be carried out inside a closed chamber. The atomizing gas was filtered using a two-stage system which included a HEPA filter for ultra fine particulate removal. This filter was measured to have an efficiency >99.97%. A laser aerosol spectrometric analysis was performed on the effluent gas downstream of the HEPA filter using a LAS 250X (Particle Measurement Systems, Inc.) during tin spray runs. The total particulate mass flow rate through the filter was measured to be 30 nanograms per second (30×10^{-9} g/s). For lead, this would correspond to a mass flow rate of 47×10^{-9} g/s. Measurements of background signals indicated that many, if not all of these counts were due to ambient dust particles.

To provide general guidelines for spray forming experiments, established operating procedures needed to be modified for spray forming lead. A copy of these procedures is given in Appendix B.

Table 5. Final set of Pb1.4Se spray runs.

INEL ID#	Substrate Material	Melt Temperature (°C)	Nozzle Temperature (°C)	Nozzle Gas Temperature (°C)	Distance from Nozzle Exit to Substrate (in)	Substrate Preheat Temperature (°C)
1J3325A-1	Polyimide	440	424	440	9	25
1J3325A-2	Polyimide	442	435	444	9	25
1J3325A-3	Plexiglass	442	419	444	9	25
1J3325A-4	Plexiglass	440	436	440	9	25
1J3325A-5	Copper	440	424	443	9	25
1J3325A-6	Copper	438	431	442	9	25
1J3335A-7	Stainless Steel	440	432	440	9	25
1J3335A-8	Stainless Steel	439	419	440	9	25
1J3325A-9	Glass	440	437	444	9	25
1J3325A-10	Glass	438	429	442	9	25
1J3335A-11	Glass	441	433	440	12	25
1J3335A-12	Glass	438	430	440	11	25
1J3335A-13	Stainless Steel	436	420	441	9	250
1J3335A-14	Stainless Steel	438	391	438	9	200
1J3335A-15	Stainless Steel	438	413	440	9	100
1J3335A-16	Stainless Steel	440	405	438	9	50
1J3335A-17	Stainless Steel	438	427	437	9	150

Notes: Nozzle pressure set at 20 psia, Crucible pressure set at 17 psia, and Substrate Speed Controller set at 50% for all runs. Material sprayed was Lead alloy (Pb1.4Se).

Appendix A

SAFETY EVALUATION for SPRAY FORMING LEAD STRIP

1.0 INTRODUCTION

This program involves cooperative research conducted with Johnson Controls, Inc (JCI), under CRADA agreement 95-CR-01. The work involves spray forming two lead alloys into strip at INEL for analysis by JCI. A total of about 16 strip samples will be produced. The work is related to strip forming activities with other metals (Al-base, Tin-base, etc.), but differs due to the toxic nature of lead.

2.0 PROJECT, PROGRAM, OR EXPERIMENT DESCRIPTION

Two lead alloys will be spray formed into strips using INEL spray-forming technology. During spray forming, fine droplets of the metals are formed as a result of the interaction of a high velocity gas jet with a stream of the molten metal. The droplets cool in flight and deposit onto flat plates (substrates) where they weld together while replicating the shape and surface texture of the substrate. The lead alloy strips will be removed from the substrate and sent to JCI for evaluation. The experiments take place in an enclosed vessel (chamber) that is purged with nitrogen gas.

3.0 PROJECT, PROGRAM, OR EXPERIMENTAL ORGANIZATION AND RESPONSIBILITY

Department Manager: Dr. James Key (525-5899)
PI/TL: Dr. Kevin McHugh (525-5713)
Senior Technician performing experiments with Dr. McHugh: Ms. Linda Wallace

4.0 EXPERIMENTAL METHOD OR PROCESS

A detailed procedure, entitled "Bay 1 Spray Forming Operation" is attached [1]. This procedure lists the sequential steps that will be followed when spray forming lead strips. A general description of activities follows.

About 10 lbs. of a lead alloy will be cut and loaded into a crucible that is sealed to a spray nozzle. A crucible lid will be sealed to the crucible using a high temperature graphite gasket. This allows the atmosphere inside the crucible to be purged free of oxygen and restricts slag formation during melting. The alloy will be heated about 100°C above its melting point (327°C) and pressure-fed (by applying a positive pressure of ~ 2 psig) into a deLaval (converging/diverging) nozzle, which is transporting a high velocity gas heated to about 427°C. The metal will be immediately transformed into fine (~25μm) droplets, entrained by the gas jet, and deposited onto glass plates that are swept through the jet to produce strips about 4" wide X 12" long X 0.020 to 0.060" thick. A pneumatic flow valve (stopper rod) is used to start and stop the flow of metal into the nozzle. After a strip is produced, an operator using the leak tight side ports of the glove box, will interchange substrates plates, adjust the appropriate experimental parameters, and repeat the spray process. In this way, many strip samples will be produced without opening the chamber or exposing personnel to lead.

It is anticipated that three spray trials will be conducted and the total amount of lead loaded into the crucible will be about thirty lbs. Of this about six lbs. of overspray particulate (lead powder) will be generated. Most of the powder will settle to the bottom of the chamber and side walls where it will be removed using the attached procedures and stored in a hazardous waste container. The rest of the overspray will be trapped within the exhaust filters which will be disposed of in a hazardous waste container. A small amount of lead will also remain inside the crucible and will be removed and disposed of with the lead powder. After all experiments are completed, the spray nozzle/crucible assembly will be disposed of in a hazardous waste container. All lead waste generated will be in the solid state either as powder or bulk material. After spray forming all lead strips with one alloy, the strips will be detached from the substrate plates, labeled, boxed, and shipped to JCI for evaluation. The process will be repeated with another lead alloy.

The compositions of the two alloys, toxicity data, and other specific data are summarized on the attached MSD sheets [2,3], see also [8]. In one, lead is alloyed with small amounts of aluminum, tin, and calcium. In the other, the alloying elements are antimony, arsenic, and selenium. Toxicity data for Sb and As are included with the MSD sheets. As mentioned, approximately 30 pounds of lead will be spray formed (about 15 lbs. of each alloy).

5.0 EQUIPMENT

The spray system used to produce lead strips consists of the modified, leak-tight glove box shown in Figure 1. It houses the main spray-forming components, i.e. nozzle, gas heater, crucible, and substrate in an inert gas environment. The system also contains a manually operated gas manifold, data acquisition computer, and associated electronics: temperature and pressure monitors, gas flow-rate monitor, gas heater interlock, and current and voltage monitors. An oxygen-level sensor can be used to monitor the atmosphere both inside and outside the chamber. A two-stage particulate filtration system removes airborne particulate (overspray powders) from the exhaust discharged to the atmosphere by a high throughput blower. The atmosphere inside the chamber is purged with nitrogen during operation. The action of the blower also maintains a slight (~.1 psia) subatmospheric pressure inside the chamber. As the attached HEPA Filter Test Report indicates, the particulate filtration system was tested 2/23/95 and passed with a percent efficiency of >99.97 [4].

6.0 SAFETY ASSESSMENT

6.1 <u>Unattended Experiments/Working Alone</u>

No unattended experiments will be performed.

The most common failure scenarios involve loss of utilities, particularly power, to the apparatus. There are no serious consequences associated with loss of utilities.

A potentially more serious scenario would involve exposure of the spray jet and overspray powder to oxygen (air). Many finely divided powders pose an explosion risk if the correct combination of particulate (particularly airborne) concentration, ignition source (e.g. a spark or flame), and oxidizer (e.g. oxygen or air) exist. In the case of lead, however, the index of explosibility is listed as <<0.1 placing its explosion potential lower than that of tin (0.1), a material routinely spray formed in our laboratory (see Bureau of Mines Report of Investigations No. 6516, attached) [5]. The relatively low explosivity of lead powder is also documented in the Metals Handbook, Vol. 7, p. 197 (see attached document) [6].

6.2 <u>Qualifications</u>

See attached resumes'

6.3 <u>Training Requirements</u>

Kevin McHugh and Linda Wallace attended a respirator training class 3/21/95 and are qualified to use a MSA full face respirator (see attached photocopy of Respirator Authorization Cards) [7].

6.4 <u>Medical Surveillance Requirements</u>

N/A

6.5 <u>Air, Personnel, and Environmental Sampling and Analysis Requirements</u>

Linda Wallace was monitored for airborne particulate exposure during spray forming runs with tin that were conducted to simulate lead strip spray runs. Results indicated..............................

6.6 Work Site Control Measures Requirements

A chamber exhaust filtration system, used to remove airborne lead particulate from the exhaust, has been efficiency tested for this application (see attached HEPA Filter Test Report). A description of other personal protection equipment is given in "Bay 1 Spray Forming Operation" [1]. Basically disposable latex gloves, labcoat, safety glasses, and MSA full face respirator equipped with HEPA filter, will be worn by personal during the handling of bulk lead, lead powder, and during spray forming experiments.

6.7 Radiation Control

N/A

6.8 Chemical Use and Waste Generation

All bulk and particulate waste generated as a result of experiments will be stored in a hazardous waste container installed at the spray forming facility. This container will be used for lead waste storage only. In addition, blades used to cut lead, paper used to collect cuttings, spent filters, crucible, and nozzle will be disposed of in the same way. After experiments are concluded, health physics personnel will analyze the conditin of the spray equipment to ensure that the spray chamber has been properly cleaned.

6.9 Criticality Safety

N/A

6.11 NEPA Documentation

See attached documentation.

7.0 REFERENCES

(1) Metals & Ceramics Department Operating Procedures: "Bay 1 Spray Forming Operation," CAP 1-5, Revision 1, May 1995.

(2) Material Safety Data Sheet for Hard Lead (Selenium Antimonial Arsenical Lead Alloy), Environmental Services Dept., RSR Corporation, March, 1993.

(3) Material Safety Data Sheet for Soft Lead (Calcium Tin Aluminum Lead), Environmental Services Dept., RSR corporation, 1993.

(4) "HEPA Filter Test Report," Internal Report issued by L. Harrison, 2/23/95.

(5) M. Jacobson, A. R. Cooper, and J. Nugy, "Explosibility of Metal Powders," Report of Investigations No. 6516, U.S. Dept of the Interior, Bureau of Mines, p.p.3-4, 1964.

(6) C. J. Dahn, "Explosivity and Pyrophoricity of Metal Powders," Metals Handbook, 9th Ed. 7 195 (1984).

(7) Photocopies of Respirator Authorization Cards for K. McHugh and L. Wallace, Issued 3/21/95.

(8) R. P. Beliles, "Toxicity of Metal Powders," Metals Handbook, 9th Edition, 7, 205 (1984).

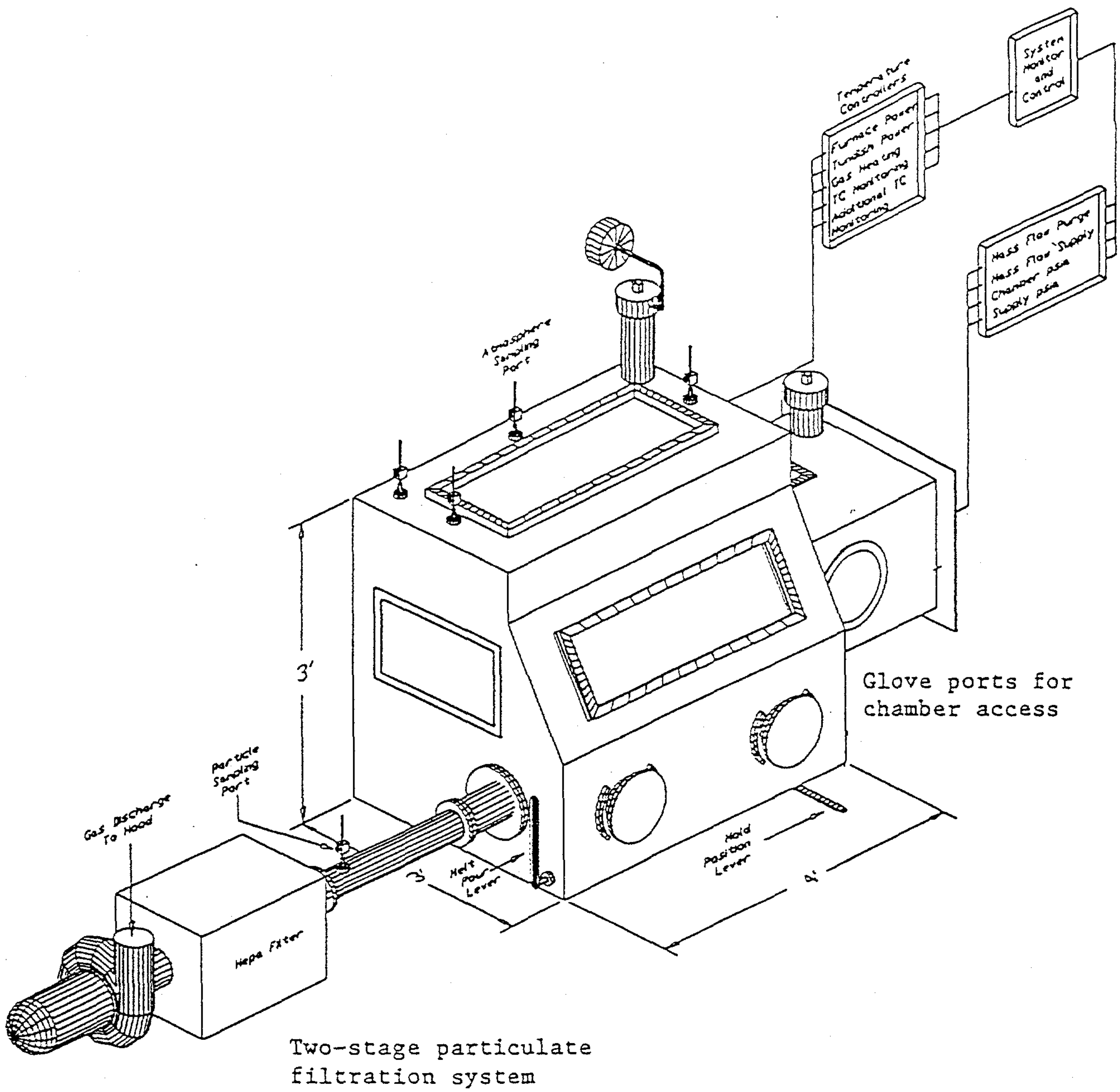
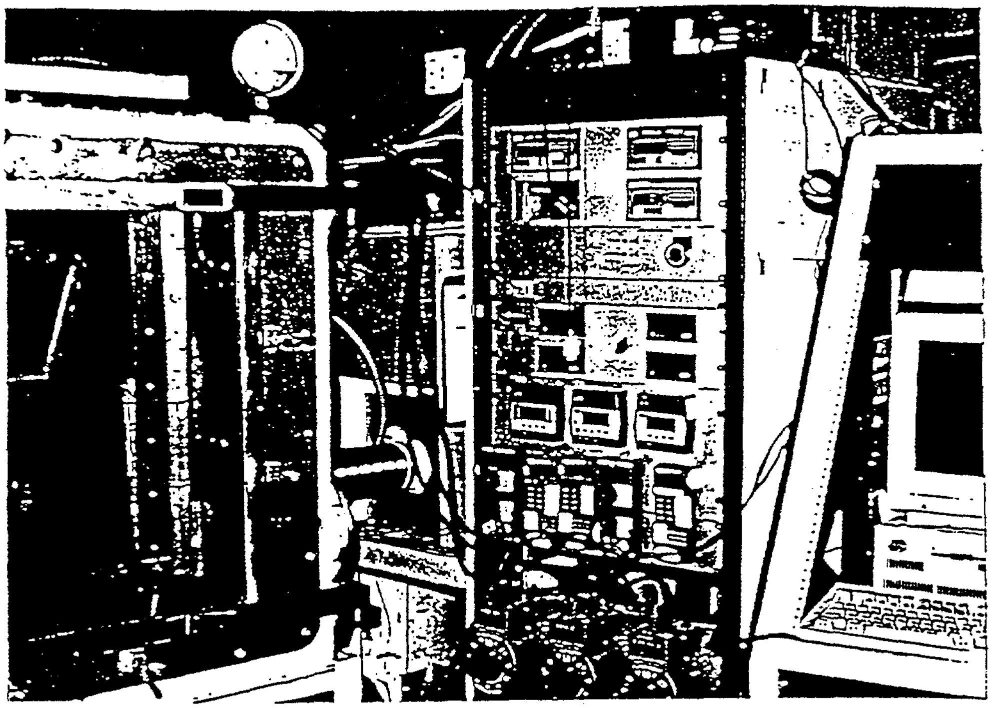


Figure 1a. Geometry of Spray Chamber



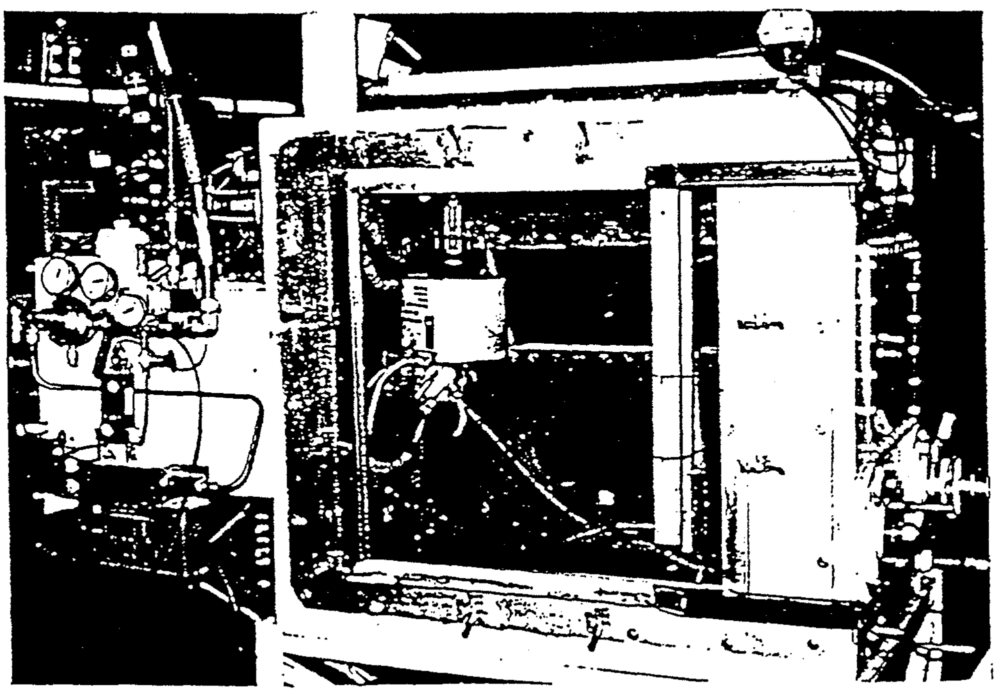


Figure 1b. Photographs of Spray Apparatus

Hazard Scenerios: (Events)

Based on the hazards identified in the checklist and the safety systems identified, using your best judgment describe the three most serious, yet realistic, hazard scenarios which can occur. (e.g. fume hood failure during methylene choloride evaporation.)

1. Over exposure to finely divided lead powder during chamber clean up.
2. Powder explosion by combination of spark source, airborne particulate and oxygen during spray forming.
3. Burns through contact with hot surfaces.

Hazard Severity:

For each of the above events decide on the hazard severity class from the descriptions below.

A. Could an event occur under normal or upset conditions which could result in death, serious injury, loss of operation of a lab module, cause of loss of $100K, a reportable emergency to DOE, or result in an uncontrolled release of hazardous waste to the environment?	Yes No	Yes = 5
B. Could an event occur under normal or upset conditions which could result in injuries, severe damage to a lab module ($10K - $100K), be reportable to DOE as an Unusual Occurrence or be reportable to the EPA or the State as a violation?	Yes No	Yes = 4
C. Could an event occur under normal or upset conditions which could result in an Off-Normal occurrence reportable to DOE, system damage between $.5K - $10K, controllable spill of a hazardous material or hazardous waste, or "No" injuries (first aid only)?	Yes No	Yes = 3
D. Could an event occur under normal or upset conditions which results in no injuries, no environmental threat, is not reportable to DOE, but some Damage to equipment may occur (less than $.5K)?	Yes No	Yes = 1

Hazard Probability:

Assess the probable frequency of the most serious failure leading to the above hazard scenarios. (e.g. for the case above fume hood failure may occur approximately monthly.)

J. **Week** - This failure could occur weekly.	Yes No	Yes = 4
K. **Month** - This failure could occur monthly.	Yes No	Yes = 3
L. **Year** - This failure could occur yearly.	Yes No	Yes = 2
M. **Decade** - This failure could occur every ten years.	Yes No	Yes = 1

Hazard Measure:

Hazard Severity x Hazard Probability = Hazard Measure

Hazard Measures

	HS	X	HP	=	HM
Hazard Scenario 1.	5	X	1	=	5
Hazard Scenario 2.	4	X	1	=	4
Hazard Scenario 3.	3	X	1	=	3

Example: Hood failure during evaporation of methylene chloride.

Hazard severity - Short term exposure of personnel to methylene chloride fumes. No injury, but requires first aid. Is an off-normal.
Hazard Severity = 3

Hazard Probability - Hoods fail several times a year so use monthly.
Hazard Probability = 3

Hazard Measure = 3 x 3 = 9

If the Hazard Measure is 5 or greater a procedure and Safety Analysis is required which minimizes the safety, health or environmental hazard. The Safety Analysis must be approved by the ISRG.

Current Applicable Procedures and Standard Practices:

1. ______
2. ______
3. ______
4. ______

Safety Evaluation Needed ☒ Yes ☐ No

ID F-5440.1A#
Ref. DOE 5440.10
(Rev. 1, 5-95)
Pg. 1

ENVIRONMENTAL CHECKLIST
U.S. DEPARTMENT OF ENERGY, IDAHO OPERATIONS OFFICE

NEPA Document No.: IFF-95-002

DIRECTIONS: Section A through D to be completed by the program/project manager. Sections E & F to be completed by the appropriate Contractor Environmental Organization (CEO), the DOE-ID NEPA Compliance Officer (NCO), or as indicated.

SECTION A. PROJECT TITLE: Spray Forming of Lead Strips

DOE-HQ PROGRAM:	EM	PROJECT NUMBER:	NA
PERFORMING ORGANIZATION:	Metals and Ceramics (4025)	DATE:	June 20, 1995
DOE PROJECT TECHNICAL MANAGER:	J. A. Yankeelove	TELEPHONE NUMBER:	526-7049
PERFORMING ORGANIZATION CONTACT:	J. F. Key	TELEPHONE NUMBER:	525-5397

SECTION B. Project Description: Attach a complete and concise description of the project or action, including type of action (e.g., new construction, process modification, maintenance, new activity, research and development, or work for others), purpose and need, pollution prevention and waste minimization measures, projected start and end dates, and approximate cost.

SECTION C. Sources of Impact: Would the action involve, generate, or result in changes to any of the following? (If yes, explain on attachment.)

Source	Yes	No	Source	Yes	No	Source	Yes	No
1. Air Emissions	x		8. Water/Well Use		x	15. Hazardous Waste	x	
2. Asbestos		x	9. Water Course Modification		x	16. Radioactive Waste		x
3. Work Force Adjustment		x	10. Pesticide Use		x	17. Mixed Waste		x
4. Excess Noise Levels		x	11. Chemical Use/Storage	x		18. Radiation Exposure		x
5. Utility Modification		x	12. Petroleum Storage		x	19. Liquid Effluent		x
6. Soil Disturbance		x	13. Solid Waste		x	20. Sensitive Resources		x
7. Water Treatment		x	14. PCBs		x	21. CERCLA/RCRA Site		x

SECTION D. The action is determined as: (check one of the following)

☐ **Appendix A Actions** Applicable Appendix A CX section: ____ Appendix A Project Manager Signature: ____
DOE-ID Approved CX: INEL-95-012

☐ **Routine Maintenance (RM)** Applicable RM CX section: ____ Certified RM Reviewer Signature:
DOE-ID Approved CX: INEL-92-030 ◊◊◊ **If RM, complete Section E.** ◊◊◊

☒ **Further NEPA documentation is required. Forward to appropriate CEO for NEPA document number and determination.**

*************************TO BE FILLED OUT BY THE CONTRACTOR ENVIRONMENTAL ORGANIZATION*************************

SECTION E. Category Evaluation Criteria: Would the action . . . (If yes, explain on attachment.)

	Yes	No
1. Require cultural, historical, or biological clearances?		x
2. Potentially impact sensitive resources identified in Item 1 above. Describe the mitigation plan.		x
3. Require or modify federal, state, or local permits, approvals, etc.?	x	
4. Be inconsistent with any existing consent orders or agreements (i.e., FFA-CO, site wide treatment plans, etc.)?		x
5. Create hazardous, radioactive, PCB, or mixed waste for which no disposal is available?		x
6. Require siting, construction, or modification of a RCRA or TSCA regulated facility?		x

SECTION F. NEPA level of documentation and reference(s).
CX: ____ EA: ____ EIS: ____ Previously Approved NEPA Documents: x Not covered in 10 CFR 1021: ____

Reference(s): Covered by IFF-95-010, "Controlled Aspiration Process Research Facility," approved by DOE-ID 4/3/95.

The proposed action would not: 1) threaten a violation of applicable statutory, regulatory, or permit requirements for environmental, safety, and health, including requirements of DOE orders; 2) require siting and construction or major expansion of waste storage, disposal, recovery, or treatment facilities; 3) disturb hazardous substances, pollutants, contaminants, or CERCLA-excluded petroleum and natural gas products that pre-exist in the environment such that there would be uncontrolled or unpermitted releases; 4) adversely affect environmentally sensitive resources. In addition, no extraordinary circumstances related to the proposal exist which would affect the significance of the action, and the action is not "connected" nor "related" (40 CFR 1508.25(a)(1) and (2), respectively) to other actions with potentially or cumulatively significant impacts.

Name: Mary B. Street Telephone No.: 526-7694

Signature: M B Street Date: 6/26/95

Metal and Ceramics Department Operating Procedure	Title: Bay 1 Spray Forming Operation Issue Date: May 1995 Number: CAP 1-5 Legend: Revision: 1

GENERAL

Purpose--The purpose of this procedure is to provide general guidelines for conducting experiments on the Bay 1 spray forming test apparatus. Because individual parts of the test equipment, and methods of test performance may vary or require modification as the test series progresses, this procedure must allow flexibility as determined by the Principle Investigator (PI).

Equipment and/or Material Required to Perform Work Scope

1. An inert gas delivery system which controls and delivers nitrogen gas at various temperatures and pressures.

2. A nozzle box assembly containing the nozzle assembly, the furnace heater coil assembly and various thermocouples to monitor gas, nozzle and melt temperatures.

3. Temperature controllers, substrate controller, and furnace variac.

4. A substrate assembly which collects the molten metal sprayed by the nozzle.

5. A two stage filtration system including a HEPA filter to remove airborn particles from gas discharged to the environment. A ventilating fan to exhaust the expended nitrogen gas through the filter to outside atmosphere and to maintain a negative pressure inside the chamber.

SAFETY ANALYSIS

The hazards of performing these experiments are:

1. Skin burns from hot objects during the melt operation.

2. Asphixiation from nitrogen gas.

3. Exposure to toxic materials when used; e.g. lead particulates.

4. Explosion of fine metal particulate.

The hazards of skin burns are minimized through the use of multiple enclosures of the molten metal, protective clothing and "Hot Surface" warning signs.

The hazard of asphixiation from inert gas is minimized through containment of the nitrogen gas by the spray system and ventilation of the gas to outside atmosphere.

Protective clothing (gloves, lab coats, safety glasses) will be worn when handling toxic metals such as lead. In addition, respiratory protection equipped with HEPA filters will be worn whenever instructed by the Area Industrial Hygienist.

Explosion of metal dust is minimized by eliminating sources of ignition, such as sparks or flames, and by conducting experiments in an inert gas-purged chamber.

Metal and Ceramics Department Operating Procedure	Title: Bay 1 Spray Forming Operation Issue Date: May 1995 Number: CAP 1-5 Legend: Revision: 1

PREREQUISITES

1. Nitrogen supply adequate for run (CAP 4-1).

2. The lead operator trained in this procedure and prior metal spray experience. Assistant operators must have read this and referenced procedures.

PROCEDURE

Conduct the test per the checklist (see Attachment 1):

EMERGENCY SHUTDOWN

If for any reason an emergency shutdown of the equipment is necessary, the following steps will put the system in a safe shutdown condition.

1. Lower stopper rod.

2. Discontinue power input to gas heaters, nozzle, and crucible.

3. Reduce nozzle pressure to 13 psia, or in case of gas leak, close gas supply valves.

Attachment 1

1. Prior to assembly, record the weight of the metal.

2. Assemble the test apparatus as directed by the PI.

3. Begin entry in Bay 1 Spray System Notebook--record unusual configuration or noteworthy items.

Initial Start-up

1. Turn on the chamber exhaust fan.

2. Check that the nozzle regulator valve is shut off on the front left side of the chamber.

3. Slowly open the main valve on the liquid nitrogen tank. (It is a double seating valve so open it all the way.)

4. At the nitrogen regulator station in bay 3, first slowly open valve 'A' then valve 330.

5. On the backside of the chamber, adjust the supply regulator to approximately 100 psig.

6. Check that all variacs are set at zero and are turned off.

7. Turn on the substrate controller.

8. Turn on the volt, amp and thermocouple meters.

Metal and Ceramics Department Operating Procedure	Title: Bay 1 Spray Forming Operation Issue Date: May 1995 Number: CAP 1-5 Legend: Revision: 1

Toxic Material Preparation

1. Spread white paper - cut lead with a hacksaw while wearing appropriate protective clothing (disposable gloves, safety glasses, labcoat, respirator).
2. Dispose of lead cuttings and blade into hazardous waste container.
3. Wear protective clothing when loading the crucible.

Preparation for Spraying

1. Fill out the data worksheet for bay 1.
3. Verify the nozzle is correctly aligned with the substrate.
4. Load the furnace with the spray material.
5. Check that the nozzle and crucible TC's are in place.
6. Mount the substrate on the translator.
7. Determine the speed of the substrate and record it on the data worksheet.
8. Determine if the stopper rod is aligned and working properly. With the chamber door closed verify the stopper rod moves up and down using the control on the outside of the chamber. Then with the stopper rod control in the down position apply pressure (approx. 13 psia) to the crucible. Apply pressure to the nozzle (approx. 20 psia). If the crucible pressure lowers the stopper rod does not have a good seal and must be adjusted.
9. Adjust the crucible pressure to 13 psia.
10. Turn on the flow to the crucible lid for cooling.
11. Turn on applicable heater variacs. Periodically check temperatures and adjust as needed.

Spraying

1. Load the data acquisition system on the PC by selecting B1OP at the main menu. To begin collecting data tap the space bar once.
2. When the furnace crucible TC registers approximately 75% of the desired temperature, adjust the nozzle regulator to at least 18 psi.
3. Turn on the emergency shutoff and heater selector switches on the Interlock Control System. Turn on the gas heaters power switches.
4. Adjust the nozzle regulator to achieve the desired nozzle inlet pressure for spraying. (The nozzle inlet pressure is indicated on pressure transducer labeled Nozzle)

Metal and Ceramics Department Operating Procedure	Title: Bay 1 Spray Forming Operation Issue Date: May 1995 Number: CAP 1-5 Legend: Revision: 1

5. The moment the melt begins to spray from the nozzle, turn on the power to the substrate translator which will move the substrate through the nozzle plume.

6. When the spray run is concluded, terminate the data collection by pressing both the control and backspace keys at the same time.

7. Shut the power off to the gas heaters and turn off all variacs.

8. Adjust the nozzle regulator to reduce the nitrogen flow through the nozzle to 50 slpm.

9. If another run is to be made, repeat this procedure beginning with the Spraying Preparation steps.

Shut Down

1. After the last run, continue to let nitrogen flow through the nozzle at the rate of ~50 slpm to cool the nozzle.

2. Once the nozzle has cooled to below 100°C, shut the nitrogen off at the main tank outside. (The nozzle regulator at the chamber should still be open to allow the nitrogen supply line to bleed.)

3. Return to the chamber and clean up the area while the nitrogen supply line bleeds.

4. Once the nitrogen flow has stopped (indicated by flow meter), go to bay 3 and close valves 'A' and '330' at the regulator station.

5. On the front of the chamber, turn the nitrogen supply valve to the off position and back off the nozzle regulator.

6. Turn off all thermocouples and digital monitors.

7. Turn off the substrate controller.

8. Shut off the exhaust fan.

Clean Up of Toxic Material

1. Don protective clothing including disposable gloves, labcoat, safety glasses and a HEPA filter respirator. The worker must be respirator trained and wear the respirator during the entire cleanup process.

2. Turn on exhaust fan.

3. With a brush collect as much overspray as possible. Store in an appropriate hazardous waste container.

4. Collect remaining overspray material with the HEPA vacuum.

5. Clean the windows on the inside of the chamber, placing soiled towels in the hazardous waste container.

Metal and Ceramics Department Operating Procedure	Title: Bay 1 Spray Forming Operation Issue Date: May 1995 Number: CAP 1-5 Legend: Revision: 1

6. After cleaning chamber remove protective clothing. Dispose of gloves in the hazardous waste container.

7. The filters in the venting system and in the HEPA vacuum, along with material collected in the vacuum, are hazardous waste.

8. Comply with Company Procedures Vol. IV, section 8.7 and ERAD Standard Practice 4.3 for the management of hazardous waste accumulation areas, for conditionally exempt generators.

Clean Up of Non Toxic Material

1. Follow the above procedures with the following changes:
A dust mask may be used in place of the HEPA filter respirator.
Recycle excess material and dispose of used items in the City's dumpster.