Materials Preparation Center

purification

preparation

fabrication

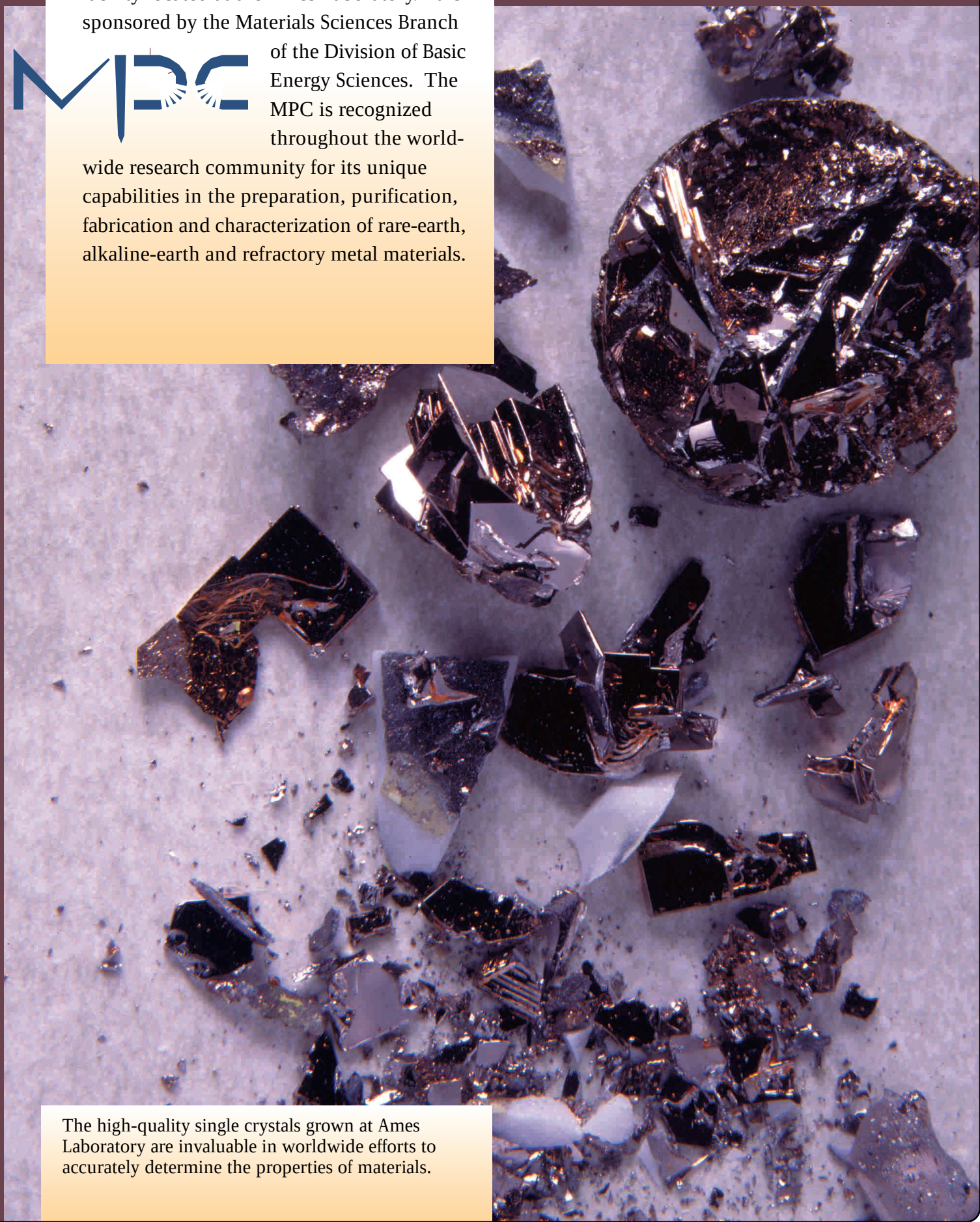
characterization

AMES LABORATORY A U.S. Department of Energy Laboratory operated by Iowa State University

Materials Are Our Business

The Materials Preparation Center (MPC)

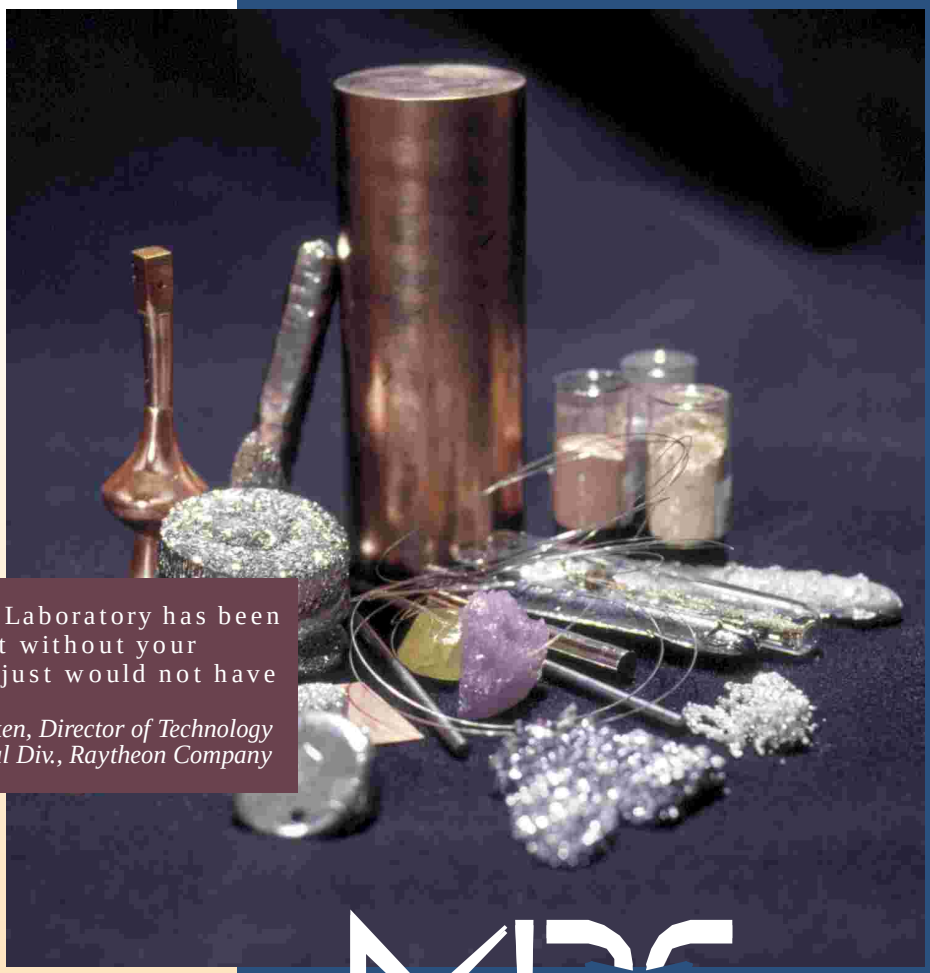
is a U.S. Department of Energy (DOE) user facility located at the Ames Laboratory. It is sponsored by the Materials Sciences Branch of the Division of Basic Energy Sciences. The MPC is recognized throughout the world-wide research community for its unique capabilities in the preparation, purification, fabrication and characterization of rare-earth, alkaline-earth and refractory metal materials.



The high-quality single crystals grown at Ames Laboratory are invaluable in worldwide efforts to accurately determine the properties of materials.

PURITY BY DESIGN

Established in 1981, the Materials Preparation Center is a one-of-a-kind user facility, which is acutely sensitive to the needs of researchers. Providing research and developmental quantities of high-purity materials and unique characterization services to scientists at university, industry and government facilities on a cost-recovery basis, the MPC allows access to novel materials and new technologies as they are developed.



The MPC is renowned for its outstanding technical expertise in alloy design, creating materials that exhibit ultrafine microstructures, high strength and high conductivity -- properties of great potential value to American technology.

"The materials support provided by Ames Laboratory has been noted repeatedly ... and it is obvious that without your organization, the progress made to date just would not have been possible."

Bradford A. Becken, Director of Technology
Submarine Signal Div., Raytheon Company

The MPC has an established reputation for close interaction with its customers, providing the kind of personal service required to meet each user's individual needs. The Center annually satisfies hundreds of requests for customized materials and services that are unavailable from commercial suppliers and unmatched in quality anywhere else in the world.

"In our search for high-purity metals, we were greatly assisted by the Materials Preparation Center of the Ames Laboratory. We have received technical information, advice and sample materials, all of which helped us to build onto the technology developed by MPC. We wish to acknowledge this assistance and thank the Department of Energy as well as the MPC for making their technolgy available to the research community."

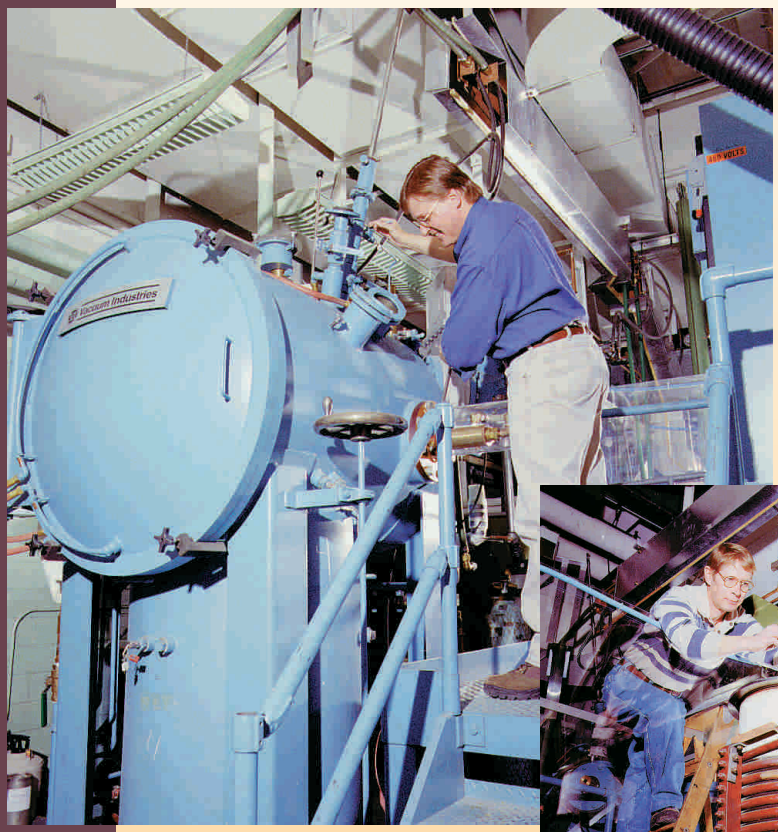
Dr. Otto Adlhart, Director of Research
AF Sammer Corporation

Pure Metals & Custom Alloys

The Materials Preparation Center specializes in the preparation, purification and fabrication of high-purity rare earth metals, refractory metals, alkaline earth metals, and alloys in single and polycrystalline forms.

Preparation and Purification Capabilities

- electron-beam melting and zone refining
- distillation and electrotransport purification
- nonconsumable and consumable arc melting/casting
- controlled-atmosphere induction melting/casting (chill casting)
- induction and resistance furnaces (several configurations)



These capabilities allow the MPC to prepare special alloy ingots, from several grams to 25 kg, under controlled conditions to maintain purity and compositional control.

Fabrication Facilities

- 100-ton, 12” rolling mill (rod and flat rolling)
- 6” rolling mill
- 700-ton hydro extrusion press
- rotary die swaging (1” - 0.014”)
- 12.5-ton, 10’ and 1.2-ton, 8’ hydraulic wire-drawing benches
- hot and cold isostatic pressing (HIP & CIP)

Mechanical Testing Capabilities

- 60 kip Baldwin compression tester
- 22 kip MTS servo-hydraulic tensile-testing machine
- 220 kip MTS servo-hydraulic tensile-testing machine
- 20 kip Instron servo-hydraulic tensile-testing machine
- 20 kip Instron screw-driven tensile-testing machine
- computer-controlled creep-testing unit (room temp. to 1000 C)

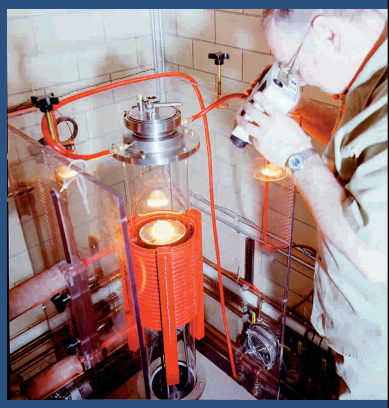
"The Ames Laboratory has provided invaluable assistance on bronze-processed and internal-tin-processed Nb_3Sn materials by supplying high-quality specialty alloy castings, unavailable commercially, in a very timely manner."

Mr. Eric Gregory,
Vice President and General Manager
Supercon Inc.

Rare-Earth Metals and Alloys

Ames Laboratory has been actively involved in the preparation of pure rare-earth metals since the early 1940s, when Dr. Frank H. Spedding and his group of pioneers developed the ion-exchange process, a technique that separates the "fraternal fifteen" plus yttrium and scandium. As a result of this early Ames Lab effort and subsequent work, high-purity oxides are available from which high-purity rare-earth metals can be prepared. In most cases the rare-earth oxides are first converted to their respective fluorides and are then reduced metallothermically on a kilogram scale, using pure calcium metal specially sublimed for this purpose. The resulting metals are very pure. Nonetheless, many are further refined at the Materials Preparation Center by one of the following processes:

- sublimation,
- distillation,
- vacuum casting,
- zone refining, and
- electrotransport processing.



Quantities of high-purity rare-earth metals and alloys in single and polycrystalline forms are available to scientists outside of the Ames Laboratory. Complete chemical analyses accompany the materials. Special preparations of high-purity alloys and compounds are also available in small quantities.

"We are absolutely delighted with the results! The neodymium is beautifully free of radioactivity, confirming that your procedure was a success."

Michael Moe
University of California, Irvine

"I am grateful to you and your colleagues at the Center for preparing such high-quality rare-earth sources. This obviously has contributed a great deal to the success of our work."

J. Raynien Kwo
AT&T Bell Laboratories

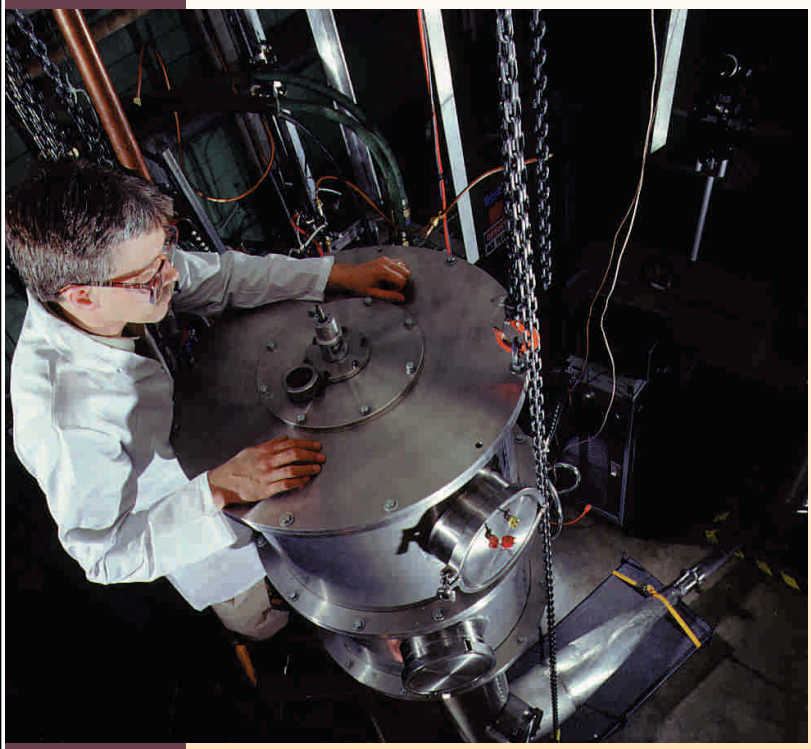
"Thank you so much for providing high-quality cerium metal for our research ... our crystals of CeNiSn have been grown from the Ames cerium."

Toshiro Takabatake
Associate Professor
Hiroshima University

Metal Powders

High-Pressure Gas Atomization (HPGA)

The MPC's high-pressure gas atomization capabilities make possible higher-quality powder materials and alloys than can be produced using current commercial atomization systems. The fine, clean, spherical shapes of the powders created with the HPGA technique allow greater compaction, maximizing the number of powder particles that can be packed into a limited space and enhancing the consolidation processes and the final properties of the product.



Consolidation facilities include:

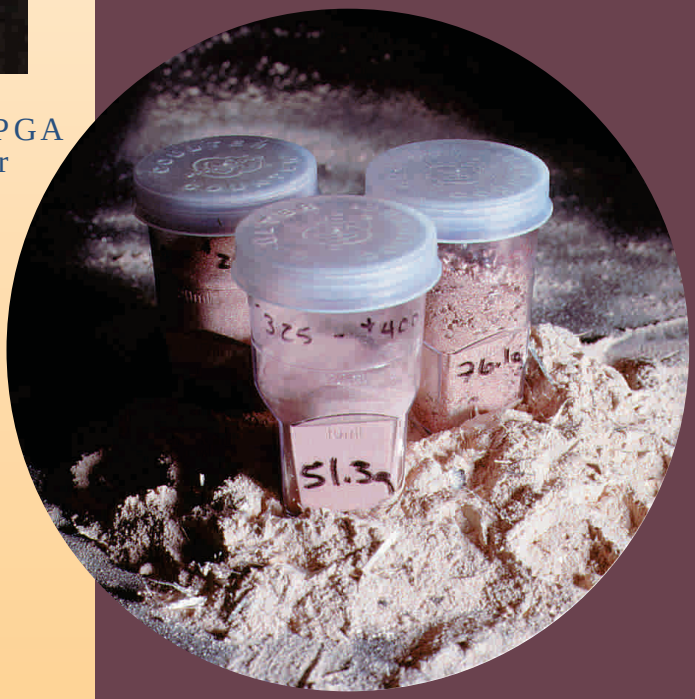
- a cold isostatic press (100 ksi),
- hot isostatic press (45 ksi, 1500 C),
- twin-screw extruder,
- single-screw injection molder, and
- sintering furnaces.

"I am very encouraged that the control in composition I desire is achievable in practice. No (other) commercial company was willing to undertake this project, nor were they able to satisfy me that they understood my requirements regarding uniformity of composition."

Dr. Tom Barbara, Senior Engineer
Varian Associates, Inc.

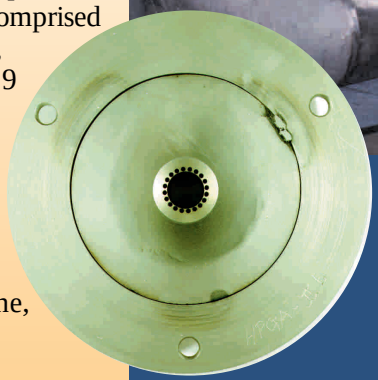
In addition to a 10 Kg-charge HPGA and a 25 Kg-charge HPGA, other MPC facilities for preparing powdered metals include:

- a centrifugal gas atomizer,
- a rapid-solidification melt-spinning furnace,
- an inductively-coupled-plasma powder unit,
- various particle-size-analysis and separation instruments, and
- a hot-outgassing/vacuum-canning station.



Ames Lab's design of this 15-foot tall atomizer and its patented nozzle is critical to the advanced properties of metal powders produced at the Lab. As molten metals flow through the nozzle, very cold high-pressure gas blasts them into extremely fine particles that cool rapidly as they fall to a collection point.

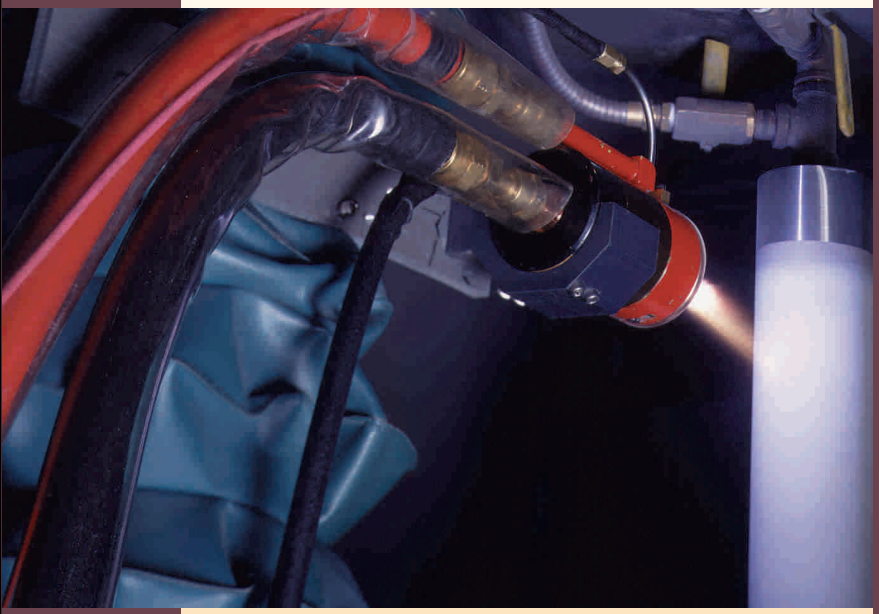
Packing the punch that delivers the powder, Ames Lab's unique HPGA nozzle is comprised of twenty miniature rocket nozzles, each with a throat diameter of 0.029 inches. Compressed gas shoots through these tiny nozzles at three times the speed of sound, blasting a stream of molten metal into extremely small droplets that cool at one million degrees per second and solidify instantaneously into fine, spherical powder particles.



Metallic and Ceramic Coatings

Plasma arc spraying is one of several thermal spraying processes that is characterized by melting of solid feedstock, such as powder, wire or rod, and propelling the molten material onto a substrate to alter its surface properties.

The Ames Lab Plasma Spray (ALPS) Facility uses Ames Laboratory's skill and expertise with materials to make new metal and ceramic coatings to provide improved wear or corrosion resistance, thermal and electrical insulation, and oxidation protection. An initial ALPS Facility activity was to develop coatings for crucible liners, which



are able to contain molten materials that are very reactive and have high melting temperatures.

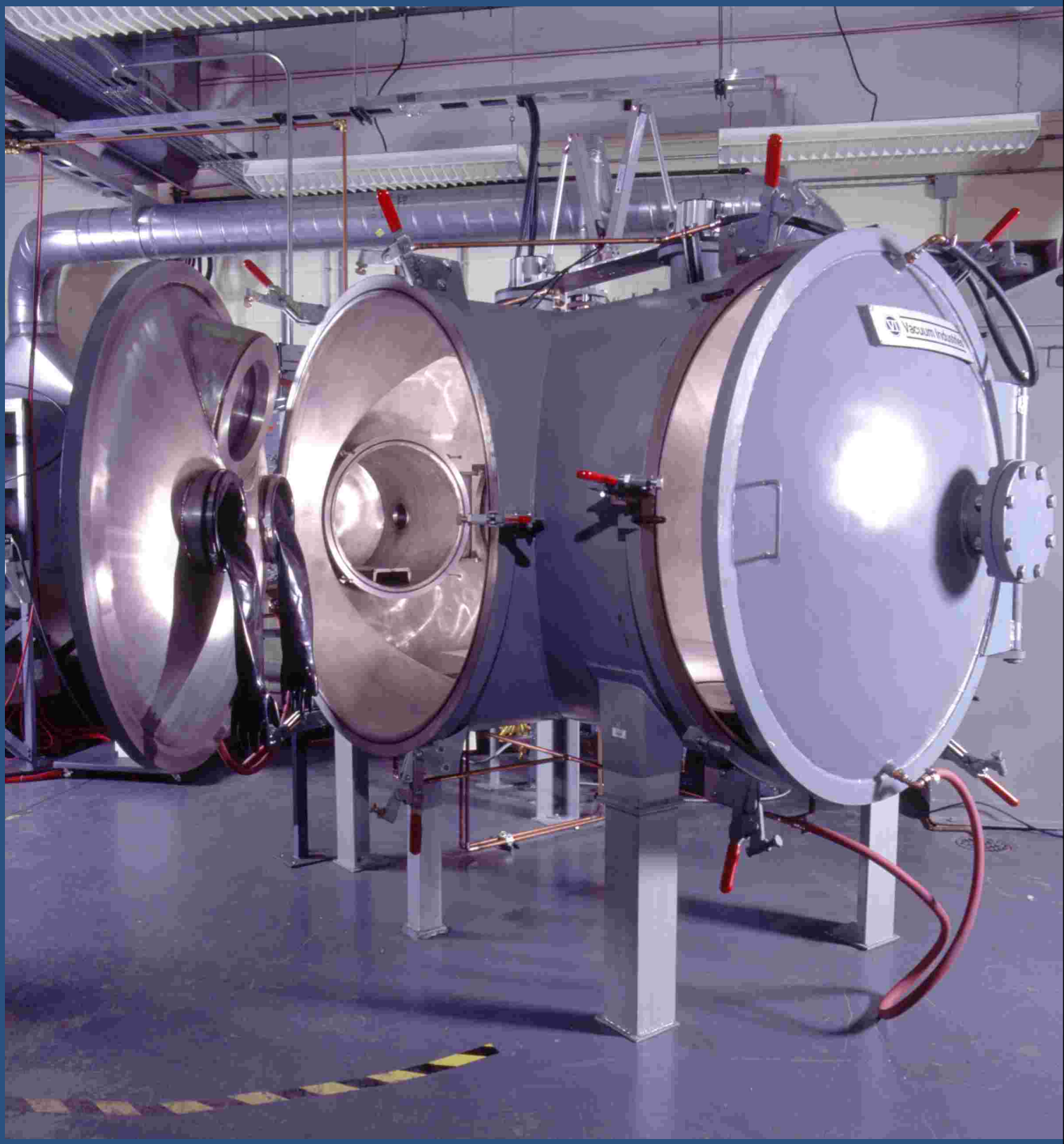
Following the early development of numerous refractory crucible-liner-coating systems, the ALPS Facility has matured in a short time to tackle new challenges, which extend the traditional capabilities of plasma arc spraying.

"The functionally graded composite coatings the ALPS Facility developed for us have been instrumental in our success with high-volume sintering of Ti-based components."

Jeffrey Moore
Advanced Forming Technologies
Longmont, CO

Examples of new materials and processes developed in the ALPS Facility as part of its research mission or in cooperation with industrial partners include:

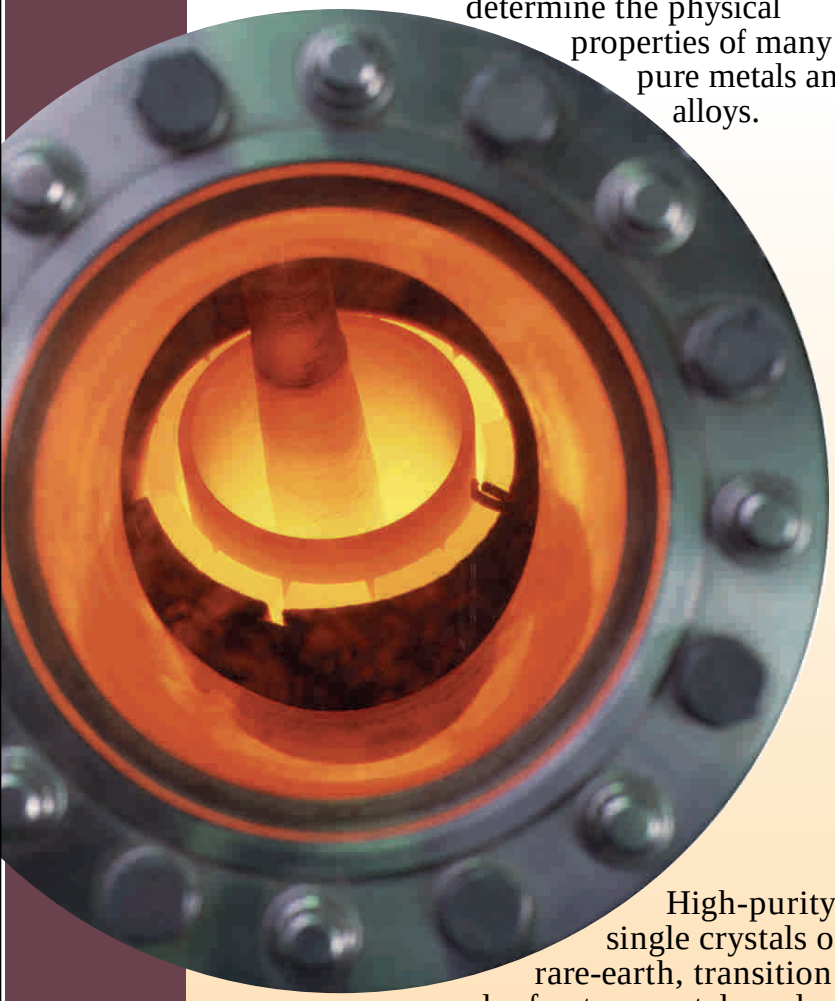
- quasicrystalline (AL-Cu-Fe) coatings for reduced friction, high hardness, oxidation resistance and low surface energy (General Motors, Deere & Co.);
- plasma spheroidization of refractory powders to produce spherical, dense particles with specific phase contents and properties (Longyear Co.);
- functionally graded materials (FGMs) involving very dissimilar materials to mitigate stress development arising from differential thermal expansion (Advanced Forming Technology); and
- fabrication of monolithic and composite free-standing shapes, which cannot be formed by conventional techniques. Variable diameter ceramic tubes with an interior layer of a refractory metal have been produced to allow rf heating of the tube.



This inert gas chamber is used for controlled-atmosphere plasma arc spraying of reactive materials.

Metal and Alloy Single Crystals

Ames Laboratory is a leader in the preparation of high-quality single crystals of rare-earth and refractory metals and their alloys. For 30 years scientists throughout the world have used crystals prepared by the Ames Lab to determine the physical properties of many pure metals and alloys.



High-purity single crystals of rare-earth, transition and refractory metals and alloys are available on a custom, best-effort basis. Some crystals can be refined to extreme purity by several metal-purification procedures, such as electrotransport and selective deoxidation. This tandem processing makes metal crystals that are among the purest in the world available for scientific research.

"Beautiful crystal ..."

A. R. Mackintosh
University of Copenhagen

MPC Techniques for Preparing Single Crystals

- arc zone melting
- cyclic-temperature technique
- float zone melting
- recrystallization grain growth
- Bridgman method
- Czochralski method
- flux techniques

MPC Crystal Fabrication and Characterization Facilities

- back-reflection Laue X-ray
- spark planing/cutting by electro-discharge machining
- diamond saws and lapping fixtures for precise orientation of crystals

"The reason that we have made such fast progress on this work was because you were able to provide us with good single crystals on such short notice. I appreciate very much the effort that you put into the single crystal growth."

Professor S. L. Sass
Cornell University

Analytical Services

The MPC's Analytical Services Group stresses those techniques which have broad capabilities and which are particularly useful for characterizing specialized materials.

Unique Mass Spectrometric Analytical Capability

Laser ionization mass spectrometry (LIMS) is used for direct analysis of solids, giving a semi-quantitative survey, including a majority of the elements from ppb to 100 percent. The LIMS technique has several advantages.

- No standard materials are necessary.
- The sample is analyzed as received, without dissolution.
- The technique is virtually nondestructive. Only 20 mg of the sample are consumed due to laser erosion of the specimen.
- Elemental coverage is essentially periodic-chart wide, except for hydrogen and the rare gases.

State-of-the-Art Capabilities

- An inductively coupled plasma-atomic emission spectrometer (ICP-AES), Thermo Jarrell Ash IRIS with CETAC ultrasonic nebulizer, provides excellent high-sensitivity and multielement analyses.
- A carbon and sulfur analyzer, Horiba Model EMIA-520, provides computer-controlled and highly sensitive analysis of carbon and sulfur in solids.
- A nitrogen and oxygen determinator, Leco TC- 436, enables computer control of time and temperature up to 3000 C in an inert gas fusion environment for the determination of nitrogen and oxygen in solids.
- Microwave-assisted dissolution with a CEM MDS-2100 can be accomplished through temperature and pressure control of twelve closed vessels for specimens that are difficult to dissolve.
- A CEM Star 6 Open Vessel microwave system is also available for efficient dissolutions of solid samples.

"Thank you for the carbon analysis. Excellent results -- very pleased."

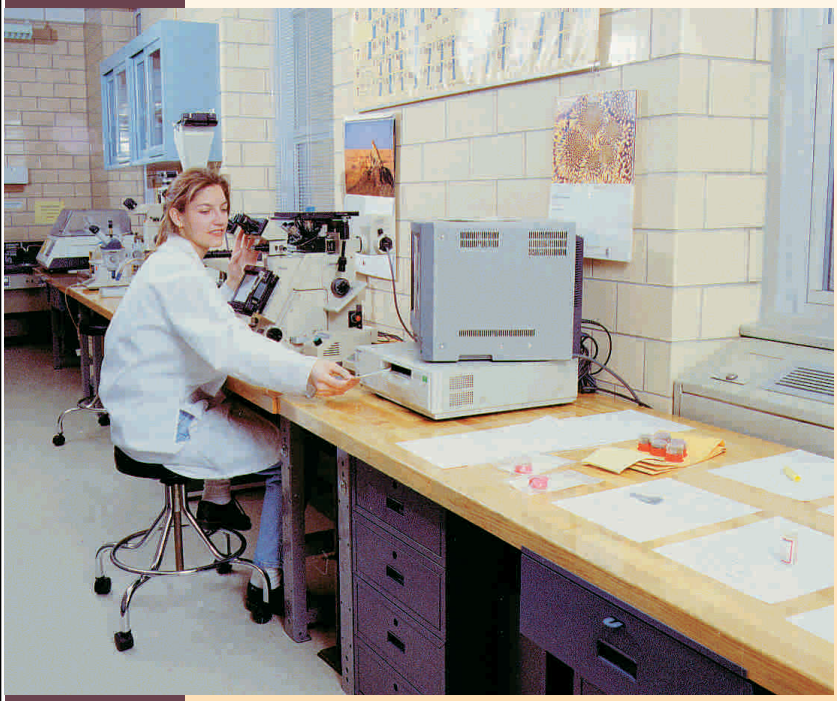
Ken Westmacott
Lawrence Berkeley Lab

Characterization Services

The MPC, through its affiliation with the Metallurgy and Ceramics (M&C) Program at Ames Laboratory, provides researchers access to a wide variety of instrumentation for microstructural and microchemical materials characterization.

Metallographic Services

- sample preparation with manual polishing, electropolishing and electrolytic etching
- image analysis
- light microscopy (brightfield, darkfield, DIC and polarized light)
- photomicro- and photomacro-photography
- hardness testing (Rockwell,Vickers, Knoop and Brinell)



Physical Properties Measurement Facilities

- differential thermal and thermo-gravimetric analysis
- differential scanning calorimeter
- magnetic susceptability (room temp. to 1K) heat capacity (350K to 1K)
- electrical conductivity (room temp. to 1000 C)
- thermal diffusivity (room temp. to 1000 C)

Surface Characterization Facilities

A PHI Scanning Auger Microprobe (SAM 660) looks at the first few monomolecular layers of materials (10-50Å) and determines the chemical composition of those layers through e-beam excitation. The SAM has the capabilities of secondary imaging to 15 keV, in-situ fracture stage, ion sputtering available for depth profiling, Auger survey mode and elemental mapping and heating stage. An energy dispersive X-ray detector to facilitate subsurface characterization is part of this system.

Microanalysis Facilities

- Phillips CM30 scanning transmission electron microscope
- JEOL 100CX scanning transmission electron microscope
- Amray 1845 scanning electron microscope

X-ray Facilities

- Philips X-ray generators with texture goniometer, 0-0 and 0-20 and powder diffractometers
- Scintag X-ray diffractometer (room temp.)

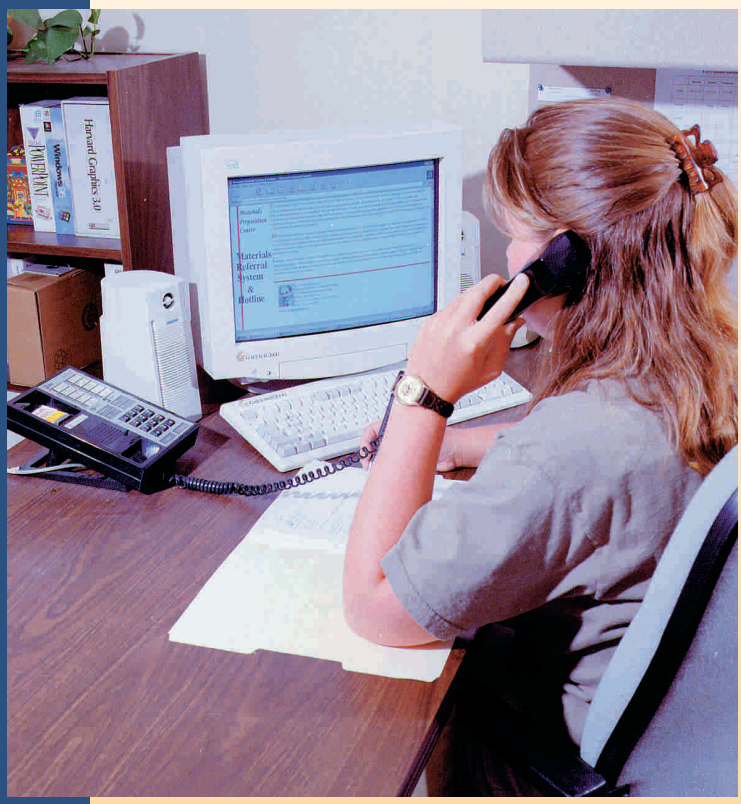
Materials Referral System & Hotline

MRSH

"I think this service is quite a find! Thanks!"

Douglas Brothers
Benedictine College

The Materials Referral System & Hotline (MRSH) offers a unique service to the general research community. By accessing our database, which now includes over 2500 companies and researchers, we are able to disseminate information about specialized materials and locate expert sources for the preparation and characterization of materials. This service is funded by the Department of Basic Energy Sciences of the U.S. Department of Energy and is provided at no charge to the user.



"I really appreciate (your) help and the friendly assistance of the referral service."

Dr. Mark E. Johnson
Los Alamos National Laboratory

MRSH is considered to be the world's leading source of single crystal suppliers and continues to build its scope of materials suppliers. Companies may list their capabilities and/or services in the MRSH database by sending brochures or product information to the address given below. All listings are free of charge.

Inquiries can be submitted to the Materials Referral System & Hotline at:

111 Metals Development Building
Ames Laboratory
Ames, IA 50011-3020

phone: 515/294-8900
800/854-1665
e-mail: ref@ameslab.gov

Procedure for Submitting Inquiries

- For each materials inquiry, please include the material name, elemental formula, and the form of material that is needed.
- For other inquiries, please include as complete a description as possible.

MPC Directory of Services

For more information about the Materials Preparation Center and how to obtain materials, please contact one of the following individuals:

Lawrence L. Jones
Director, Materials Preparation Center
121 Metals Development Building
Ames Laboratory
Ames, IA 50011-3020
phone: 515/294-5236
FAX: 515/294-8727
e-mail: jonesll@ameslab.gov

Trevor M. Riedemann
Manager, MPC Rare Earth Materials Section
123 Metals Development Building
Ames Laboratory
Ames, IA 50011-3020
phone: 515/294-1366
e-mail: riedemann@ameslab.gov

Thomas A. Lograsso
Manager, MPC Crystal Growth Section
111 Metals Development Building
Ames Laboratory
Ames, IA 50011-3020
phone: 515/294/8425
e-mail: lograsso@ameslab.gov

Analytical Services
292 Metals Development Building
Ames Laboratory
Ames, IA 50011-3020
phone: 515/294-5380

Materials Referral System & Hotline (MRSH)
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phone: 515/294-8900
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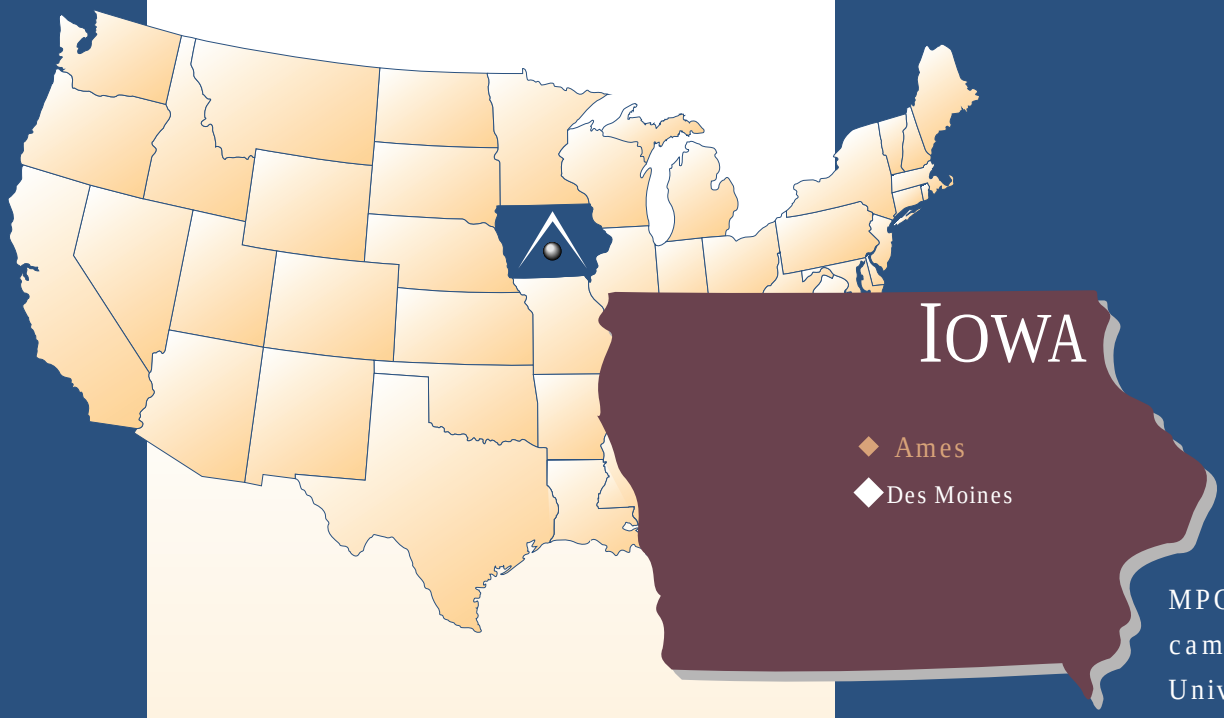
e-mail: ref@ameslab.gov

http://www.mpc.ameslab.gov

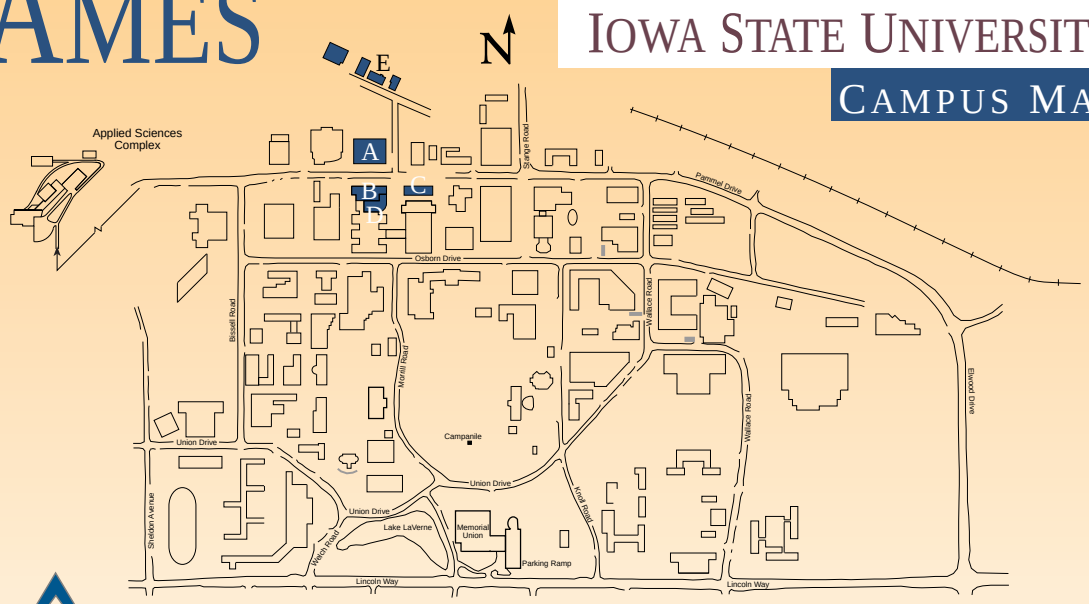
MPC Locational Map



MPC is located on the campus of Iowa State University in Ames, Iowa. Ames is 35 miles north of Des Moines.

Ames

Iowa State University Campus Map



Ames Laboratory buildings

A Metals Development (Materials Preparation Center)
B Spedding Hall
C Wilhelm Hall
D Technical and Administrative Services Facility (TASF)
E Warehouse and Shops

Visitor parking permits are available through Human Resources, 105 TASF, 515-294-2680.

Ames Laboratory is a U.S. Department of Energy laboratory seeking solutions to energy-related problems through the exploration of chemical, engineering, materials, mathematical and physical sciences. Established in the 1940s with the successful development of the most efficient process to produce high-purity uranium metal for atomic energy, Ames Lab now pursues much broader priorities than the materials research that has given the Lab international credibility. Responding to issues of national concern, Lab scientists are actively involved in innovative research, science education programs, the development of applied technologies and the quick transfer of such technologies to industry. Uniquely integrated within a university environment, the Lab stimulates creative thought and encourages scientific discovery, providing solutions to complex problems and educating tomorrows scientific talent.