



5-1996

Design and Implementation of a World Wide Web Server for the Diffraction and Thermo physical Properties Group at Oak Ridge National Laboratory

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**Design and Implementation of a World Wide Web Server
for the Diffraction and Thermophysical Properties Group
at Oak Ridge National Laboratory**

**Robert D. Bird, Jr.
Senior Honors Project
May 3, 1996
Dr. Edward S. Clark, Advisor**

The World Wide Web is one of the most interesting phenomena to invade modern society in recent years. What once, a mere two years ago, served as a method for storing and linking documents in the world of academia, is now a living and breathing commercial entity. Using a computer and a modem which can be obtained for as little as a few hundred dollars, and paying as little as ten dollars per month to an Internet provider, people can access corporate profiles and catalogs, apply to colleges, purchase anything from computers to clothing, publish their own resumes or artwork or literature or what they had for dinner last night, or find ten thousand phone sex numbers. The content of the World Wide Web is limited only by the imaginations of the people who create the pages which populate the Web. The key point, however, is that **anyone** willing to pay that ten dollar per month fee can become a Web publisher.

How does the World Wide Web provide all of this “information” to the “surfers” who have taken such a shining to it? The actual workings of the Web are transparent to most of its users. The programs that people use to access the Web, such as Netscape, Mosaic, or Lynx, are known as *clients*. They accept input from the user, which is then transported from the user’s computer to a remote computer known as a *server*. The server sits and waits for requests, and when it receives one, either sends out the requested information to the client, sends a message that it is too busy to answer, or

sends an error message which states that the requested information is not present on that particular server. The two computers communicate using a language called HTTP, or Hypertext Transport Protocol. Because many different types of computers can speak this “language”, the type of computer on either end of the connection is usually not an issue. Using this protocol, the server can send various files to the client, such as text, images, sound, and video data. The client then interprets this data in a manner which is appropriate for the particular computer it is running on.

By far the most common type of data transferred over the Web is formatted text. The standard formatting for text on the Web is called Hypertext Markup Language (HTML). This language basically consists of text which is modified by special markings, or tags, set in <brackets>. There are many different available tags for use in HTML. An extensive list may be found at <http://www.ncsa.uiuc.edu/demoweb/html-primer.html>, the Web address of “A Beginner’s Guide To HTML”, an introductory document put together by the University of Illinois. An example of a markup tag (boldface type in this case):

`This Text Is Bold`

appears on the Web as

This Text is Bold

Other tags include headers, titles, blinking text, and most importantly, links. Text which is marked with a link tag may be selected on the page,

usually by clicking on it with the mouse. Links may take the user to any other document on the Web. A link could show the user a picture or more text located on the same server as the original page, or it could show him or her a video clip from somewhere in France. The possibilities of interconnecting related documents are endless on the World Wide Web.

Obviously, since the World Wide Web is so inexpensive to use both for viewing and for publishing, and because millions of people are viewing Web pages every day, it makes sense for commercial entities to want to make use of it. Its low cost and high theoretical audience base make the Web a cheap, potentially valuable form of advertising.

The Diffraction and Thermophysical Properties Group at Oak Ridge National Laboratory (ORNL) in Oak Ridge, Tennessee is part of the High Temperature Materials Laboratory User Program. This means that any university or business in the U.S. can apply to do research at the Laboratory, and they may use the resources, including the instruments and staff, of the lab to further their own interests. This program is open to universities at no cost, and a set hourly cost for businesses. Obviously, the User Program could benefit greatly from increased advertising, especially since funding from the government is becoming harder and harder to come by. My project was to design and implement a "presence" on the World Wide Web for the Diffraction and Thermophysical Properties Group.

The first step in establishing a Web presence was to secure a computer which could be dedicated to the task of serving information to the world. The best available machine in the Group which was not being put to heavy use was a Macintosh IIfx. While this computer was several years old and somewhat outdated, it had plenty (20 megabytes) of RAM memory and a fairly large (512 megabyte) hard disk, which are two of the most important qualifications for a Web server. The other important qualification is a fast Internet connection, which was already present in ORNL's extensive computer network.

Once the computer was available, Web server software needed to be obtained and installed. The software which was chosen was WebSTAR from StarNine, Inc., the leading Macintosh Web software. This program has a distinct advantage over others in its field in that it is extremely easy to set up and configure. Because we were not planning on doing complex things like credit card transactions over the Internet, several configuration options did not have to be changed from the default. WordPerfect word processing software was also purchased for page building, though it was later noted that it was quicker to use a standard text editor, because HTML documents are just ASCII text files.

Once the server software was set up, the task of page building began. Several existing paper brochures and booklets detailing various aspects of the program were adapted for use on the server. Because there is no

monetary limit to the amount of information you can serve on a Web site, much of this information was expanded, and many pictures and icons were added to help improve the appearance of the site. Some pictures were taken directly from electronic archives, while others required scanning and/or editing before being added to the pages. A bold blue background with the acronym "HTML" (for High Temperature Materials Laboratory) was adapted from a poster design and implemented as the default background pattern for all of the pages (this background does not show up in the printouts which are included in this report, but can be seen when actually viewing the site). Colors were continually adjusted until a set which was easily viewable on just about any monitor was discovered. This turned out to be the dark blue background with white text and yellow links to click on, while previously viewed links are a light green color.

The final portion of the project was that of administration of the server. A system needed to be devised so that the Group could receive feedback from interested parties. This problem was solved by the addition of electronic mail server software. The software used for the site was Apple Internet Mail Server, a free program available from Apple. This software, in combination with special clickable links on the various pages, allows viewers to send email directly to the person in charge of the particular item they are viewing. It also saves a copy of the mail message in a file so that the mail received by the group via the Web can be stored in

a central place for later analysis. The monthly log files for the Web server software are also to be analyzed. These logs keep track of every access of every page on the site, so that the Group can know who is viewing their pages and if they possibly need to adapt the content of the pages to draw the desired audience to the User Program. As this is an ongoing task, this project only included the design and implementation of the site, while analysis and adaptation of the results will have to be dealt with at a later date.

The World Wide Web is an effective means for publishing and broadcasting information to a large number of people for a low cost. Anyone with the desire to publish on the Internet can do so with very little trouble. Hopefully, the Diffraction and Thermophysical Properties Group will benefit from the effort put into this project.

Web Server Pages

Diffraction and Thermophysical Properties Group



Welcome to the home page for the Diffraction and Thermophysical Properties Group, which includes the X-Ray Diffraction User Center, the Residual Stress User Center, and the Physical Properties User Center, located in the High Temperature Materials Laboratory (HTML) at Oak Ridge National Laboratory in Oak Ridge, Tennessee.

Visit our User Centers:



[Diffraction User Center](#)



[Physical Properties User Center](#)



[Residual Stress User Center](#)



Find what you're interested in on our [Instrument List](#).

Wow!

Look at some [highlights](#) of recent HTML User Program projects.

Find out how to [apply for the HTML User Program](#)
[About this server.](#)



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Instruments in the Diffraction and Thermophysical Properties Group

Diffraction User Center:

- Room-Temperature X-Ray Diffractometer
- High-Temperature X-Ray Diffractometer
- High-Temperature Neutron Powder Diffractometer
- High-Temperature Synchrotron Powder Diffractometer

Physical Properties User Center

Thermal Analysis:

- Simultaneous Thermal Analysis
- Differential Scanning Calorimeter/Mass Spectrometer
- Dual Push-Rod Dilatometer
- Concurrent Thermal Analysis
- Pycnometer

Thermal Transport:

- High-Temperature Laser Flash Unit
- Room-Temperature Xenon Flash Unit
- Longitudinal Bar Thermal Conductivity Cryostat
- Scanning Thermal Conductivity Microscope
- Infrared Camera
- 3-Omega

Residual Stress User Center:

X-Ray Residual Stress Facility:

- 4-Axis Goniometer/18kW Rotating Anode X-Ray Source
- 4-Axis Goniometer/2kW X-Ray Tube
- Neutron

Neutron Residual Stress Facility

[Neutron Diffraction Macro Residual Stress Mapping Facility](#)
[Neutron Powder Diffraction Micro Residual Stress Facility](#)



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HTML User Program Highlights

Residual Stress User Center

Feb. '96 - HTML Helps N.C. State Understand Adherence of Nitride Coatings to Alloy Substrates

North Carolina State University staff benefitted from the Residual Stress User Center to characterize the residual stress and texture in two hard coatings, ZrN and TiN, on Ti, Fe and Ni based alloys. Such coatings are widely used by engine companies to improve wear resistance and reduce probability of crack formation. Both sets of coating were found to possess an equibiaxial stress state with large compressive residual stresses of -1 to -3 and -3 to -5 GPa, respectively. The TiN coatings exhibited a strong (111) fiber texture while the ZrN coatings were essentially random. The stresses in ZrN were observed to decrease from -2.5 to -1.2 GPa after an annealing treatment. These data will be combined with other information to develop a model for the adherence of these coatings to alloy substrates.

Feb. '96 - Alternative Plans for NRSF Development being Evaluated

A proposal for a significant upgrade of HFIR was proposed by ORNL to the BES Advisory Committee (BESAC). The proposal, rated it #1 priority, includes construction of a thermal neutron guide hall at the location previously identified for the Neutron Residual Stress Facility and three other instruments. If funded the guide hall would have two stations dedicated for residual stress measurement under the HTML/HFIR cooperative effort. Each station would have higher thermal neutron flux and room for optimum monochromator crystals. To avoid the possibility of loss of investment if the guide hall were funded, new options for locating a dedicated NRSF are being evaluated.

Jan. '96 - EWI uses neutron residual stress facilities for study of welding of aluminum plate

Edison Welding Institute (EWI) used the neutron residual stress mapping facility to investigate the residual strain distribution in a multi-pass aluminum weld joining two half inch thick plates. The objective was to use the experimental data to provide guidance for improvement of a finite element method (FEM) model which predicts distortion and residual stress. Due to the use of undermatching filler metal and the metallurgical changes in the heat affected zone and fusion zone during welding, there are considerable uncertainties in computational models which predict plate distortion and residual stress distribution. Neutron diffraction strain measurements confirmed the qualitative characteristics of the strain distributions predicted by EWI's finite element calculations. A predicted double peak structure of longitudinal residual strain across the weld was clearly observed. In addition, the experimental data show a strong through the plate strain

dependence, which was also predicted by the finite element model. Quantitative agreement has not yet been achieved indicating that the temperature dependent thermophysical properties are not yet fully modeled. Upon refinement, EWI expects the model can be used to address welding problems in aluminum alloys, which are candidates for weight saving materials in future transportation systems.

Jan. '96 - Pre standards Meeting on Neutron Residual Stress Methods Held

A Versailles Advanced Materials and Standards (VAMAS) Technical Working Area inaugural meeting was held in France to launch development of recommended accurate and reliable procedures for making neutron residual stress measurement in crystalline materials. Eighteen attendees, plus six via FAX and e-mail, contributed to the meeting's success. Participants were primarily from Europe, with one each from Canada and Japan and three from the USA including Dr. S. Spooner of the HTML/HFIR Neutron Residual Stress Facility. This number represents a considerable growth in the method in the last couple years with new centers being established at many reactor facilities. The Technical Working Area goals, when achieved, will assure wide spread acceptance of this new technique and thereby enhance materials processing and fitness for service.

Dec. '95 - HTML Assists Rodel, Inc., In Understanding Chemical Mechanical Polishing (CMP) of Thin Films

A user from Rodel, Inc., applied the facilities of the Residual Stress User Center to study the effects of Chemical Mechanical Polishing (CMP) on thin layers of W and Al-Cu interconnect structures on Si wafers. CMP reduces the surface roughness and permits smaller line width interconnects to be successfully patterned. Residual stresses in thin layers and interconnects are believed to be responsible for defect development and ultimate failure in integrated circuits. The residual stresses, measured in the 1 μm thick W layer by X-ray diffraction methods, were found to be biaxial in nature. The stresses were highly tensile (>1 GPa) in all samples, but varied with surface treatment. The results indicate the need for further studies to develop fabrication and polishing techniques which would bring the stresses to lower levels.

Nov. '95 - HTML Assists Quantum Peripherals Colorado, Inc., to Develop Advanced Computer Memory Technologies

A user from Quantum Peripherals Colorado, Inc., visited the HTML Residual Stress User Center to determine the residual stress and crystallographic texture of Ni-19wt.%Fe and Ni-16wt.%Fe-3.5wt.%Mo thin films on Si wafers as a function of film thickness. X-ray characterization is critical to understanding the magnetic properties of these films. "Intrinsic" film stresses were measured in the 800A films via the sine-squared-psi technique and were found to be nearly 1 GPa compressive. Pole figure measurements revealed a relatively strong $\langle 111 \rangle$ fiber texture normal to the film plane in all specimens as well as a weak $\langle 100 \rangle$ texture which decreased as film thickness increased from 150A to 800A. This

information will be correlated with magnetic property and TEM measurements in an effort to understand and manipulate deposition conditions to obtain films with enhanced magnetic properties.

Physical Properties User Center

Feb. '96 - High Speed, High Resolution IR Camera Predelivery Testing Completed

PPUC staff visited Amber Inc. to receive training and perform pre shipment inspection of a Galileo infrared camera. Performance of the camera was found to be better than the specification and the camera has been shipped to ORNL. The camera will be used to map the thermal diffusivity of components, such as combustors, blades, vanes, pistons, heat exchanger tubes, and test coupons. In addition, the camera may be used to measure emissivity and for NDE studies. The camera was purchased using OIT funds in support of the ATS program and will be available part-time to users as part of the HTML User Program.

Dec. '95 - Ford Characterizes Thermal Properties of Metal Matrix Composites

Researchers from Ford Research Laboratory used the scanning thermal conductivity microscope and xenon flash thermal diffusivity system to study metal matrix composites loaded with ceramic particles. The matrix material is copper or aluminum and the ceramic particles are SiC from various vendors. Ford is studying the effect of processing conditions and thermal cyclic fatigue on the thermal conductivity of these composites. These high thermal conductivity composites are under development for possible use in electric cars.

Dec. '95 - Advanced Laser Flash Thermal Diffusivity System Operational

The Physical Properties User Center has taken delivery of, installed, and tested the multi-station thermal diffusivity instrument. This new state-of-the-art system is capable of measuring six specimens during one furnace run. Specimens may be measured in high vacuum, inert gas, oxidizing or reducing atmospheres. Special sample holders are provided for molten metal measurements up to 1700°C. The maximum operating temperature of the system is 2500°C. This instrument will be used by U.S. industry to study the effect of processing and environmental conditions, microstructure, and composition on the thermal transport properties of high performance materials.

Dec. '95 - Thermophysical Properties of NiCr alloys Determined for Casting Models

CMP Industries, a small company that produces NiCr alloy rods using a cast iron

permanent mold casting process, has used the instruments of the Physical Properties User Center to determine the thermophysical properties of three of their NiCr alloys. Their current process results in an unacceptably high defect (centerline shrinkage porosity) level of 19%. Labor costs are increased significantly because defective pieces must be sorted out and remelted. Measurement of the thermophysical properties at HTML will allow the simulation of the casting process using a commercial casting model. Process parameters will be manipulated in the simulations to optimize the casting process and reduce the porosity generated during solidification.

Dec. '95 - Successful Demonstration of New Technique for Testing Thermal Conductivity

The Physical Properties User Center successfully tested thermal conductivity (from RT to 300°C) of NASA samples using the new 3-omega system. The 3-omega technique is a new method of measuring thermal conductivity of glass and ceramics, especially coatings. NASA, the first user to provide samples, is interested in the thermal properties of Macor and glasses above room temperature. The HTML has shown the 3-omega technique to be capable of providing such information. NASA is very enthusiastic about continuing this study.

Nov. '95 - HTML Develops Capability to Determine Density of Molten Metals

Staff of the Physical Properties User Center have successfully demonstrated the capability to measure the density of a molten metal. Development of this new capability is in response to five industrial proposals and additional inquiries for the thermophysical properties of materials near and above their melting points. This new capability was developed by modifying the existing dual-push rod dilatometer and was first used to determine the density of an aluminum-silicon casting alloy to 200 degrees C beyond its melting temperature. The density as a function of temperature and the volume change associated with the liquid/solid phase transition are important parameters required as input for modeling of solidification processes including casting, semi-solid forming, and welding.

X-Ray Diffraction User Center

Feb. '96 - Washington University Uses HTXRD to Characterize Quasicrystalline Alloys

TiZrNi quasicrystals, which have potential applications as hydrogen storage materials, were investigated in collaboration with Washington University. The phase evolution from melt-spun quasicrystals and amorphous alloys were conclusively determined using high temperature XRD. The phase equilibria data are important for determining the maximum stability temperature of the quasicrystal during dehydrogenation. In addition, HTXRD was used to monitor the quasicrystal lattice contraction during dehydrogenation, and future user

proposals were planned to measure the hydrogenation/dehydrogenation kinetics and diffusion coefficients.

Jan. '96 - Crystallization of Sol-gel Coatings for Fibers in Composites Characterized

A user from the University of Tennessee studied the crystallization of mullite and aluminum titanate prepared by the sol-gel method. The oxide coatings are candidates for coating fibers in continuous fiber ceramic composites. In-situ, high temperature x-ray diffraction was effective for determining the crystallization temperatures and the reaction mechanisms leading to the formation of the complex oxides. Use of the oxides may prove effective for improving the fracture toughness of Nicalon/SiC composites and for oxidation resistance in oxide-oxide composites. Results were presented at the ACerS Engineering Ceramics meeting in Cocoa Beach, Florida this month.

Nov. '95 - High Speed X-Ray Diffraction Used to Characterize Glass Ceramic Crystallization

A user from Virginia Tech University studied the crystallization of Ba-Ge-Ga-O glasses using the high-speed, high-temperature X-ray diffraction facilities at HTML. Crystallization of glasses to form single phase, fine grained ceramics with high fracture toughness and chemical durability is critical for many applications of glass ceramics. For infrared transmitting applications, including missile domes, the Ba-Ge-Ga-O glasses show great promise. Three glass compositions were studied, all of which have excellent optical and chemical properties as compared to the commonly used MgF₂. Glass ceramic crystallization could lead to superior mechanical properties. Powder patterns were collected using the position sensitive detector; typical isothermal patterns were collected for 1 min each, with a pattern collected every other minute. These data showed that two glasses crystallized as single phase ceramics, and optimum heat treatment conditions were determined. Single phase ceramics are desirable to minimize scattering losses.

Other Highlights

Feb. '96 - LoTEC Fellow Pursues Understanding of Influence of Processing Variables on Two NZP Materials

Low thermal expansion NZP (Na Zr₂ P₃ O₁₂ and its isostructural compositions) have a unique crystal structure which permits almost unlimited ionic substitutions. Two of these NZP ceramics, BS-25 (Ba_{1.25} Zr₄ P_{5.5} Si_{0.5} O₂₄) and CS-50 (Ca_{0.5} Sr_{0.5} Zr₄ P₆ O₂₄), have potential for use in exhaust port liners for diesel engines. The cost of the final product is largely dictated by the powder synthesis and processing. Compared to the conventional solid-state oxide reaction synthesis of powders, alternative processing techniques exist which could result in significant reduction in time and cost. However, NZP ceramics are quite sensitive to compositional purity and powder characteristics, which derive from the

synthesis technique.

Under an HTML Fellowship LoTEC is characterizing powder properties essential for selecting the most suitable low cost processing method. Currently, six powders are being evaluated (four of CS-50 and two of BS 25). Powder characterization performed at HTML to this point includes: 1) SEM with EDS to determine particle size and shape, and also to inspect for elemental impurities; 2) X-ray diffraction to determine powder purity, phase content and lattice parameters (both at ambient and elevated temperatures); and 3) STA and DSC to study phase stability. In the end, results of all testing and analysis will enable LoTEC, Inc. to identify promising synthesis and processing routes for large volume production of NZP ceramics such as BS-25 and CS-50.



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HTML User Program Application Process

The process for applying to become a user at the High Temperature materials laboratory is a simple one.

- First, get a good grasp on what project you would like to work on and the requirements (instruments, time, manpower) required.
- Second, get in contact with one of our staff members (email, US Mail, and phone numbers are provided below) who you think you might be interested in working with on your project.
- Next, download and/or print, fill out, and return the proposal form to the High Temperature Materials Laboratory (details are included in the form).
- User agreement?

That's all there is to it. Once your application is received, it will go through a review process (conducted once per quarter) and you will be contacted with more information.

Contact information:

[Physical Properties User Center](#)

[Residual Stress User Center](#)

[X-Ray Diffraction User Center](#)



[Back to HTML User Centers Home Page](#)



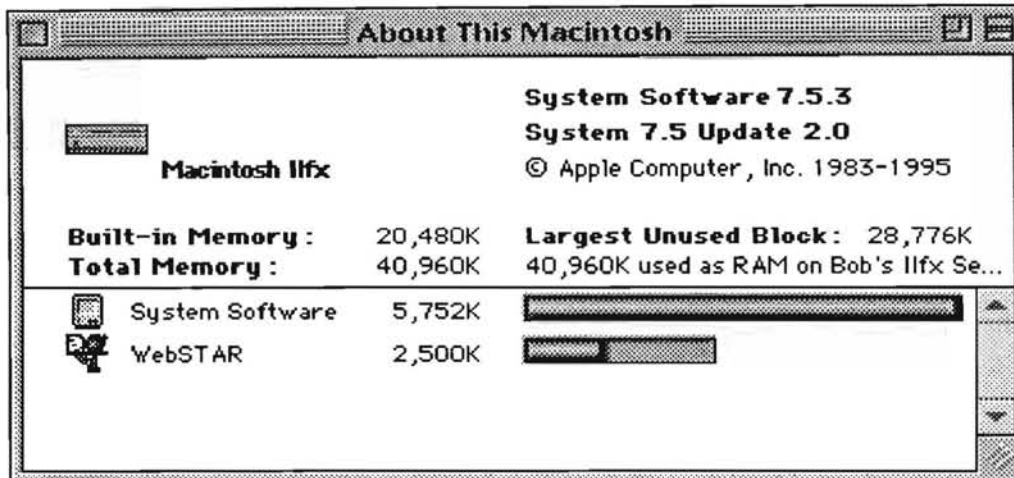
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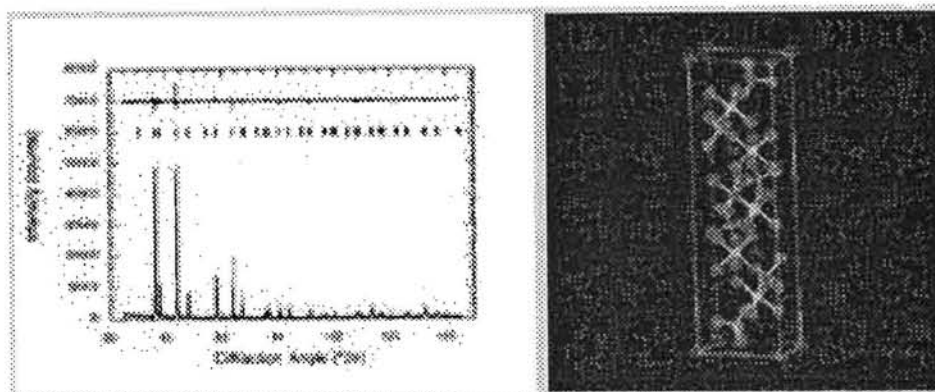
This server is running on a semi-dedicated Mac IIfx running MacOS 7.5.3. It's not the fastest machine around (by a long shot), but it does at least have plenty of memory. The server software is WebSTAR 1.2.5 from [StarNine Technologies](#). Mail is handled by [Apple Internet Mail Server 1.1](#). Pages are designed using [WordPerfect 3.5 for Macintosh](#) with built in HTML tools. Graphics are prepared using [Adobe Photoshop 3.0](#). This server is under construction and probably will be for quite a while. Please mail any comments or suggestions to me at the address below.



[Back to Diffraction and Thermophysical Properties Home Page](#)

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DIFFRACTION USER CENTER



Click on either picture to bring up a larger image

The X-ray Diffraction User Center includes both room temperature and furnace-equipped diffractometers. The high-temperature diffractometer is a theta-theta design (specimen remains horizontal at all times) diffractometer with a specimen-heating furnace capable of operation at 3000 K and a position sensitive detector for high speed measurements. The X-ray furnace is used for studies of materials properties at temperatures up to 2700 degrees C in vacuum and up to 1500 degrees C in air. Temperatures up to 1700 degrees C in nitrogen have been achieved.

In x-ray diffraction, polycrystalline solid or powdered specimens are placed in a monoenergetic, collimated beam of x-rays. Appropriately oriented grains diffract the x-rays into a detector. By sweeping the angle of incidence and detection, a spectrum of diffraction peaks corresponding to the crystal lattice spacings is produced. The lattice spacings are compared with spacings of known compounds to identify the crystalline phases.

For more information, select the links below.



[Room-Temperature X-ray Diffractometer](#)



[High-Temperature X-ray Diffractometer](#)



[High-Temperature Neutron Powder Diffractometer](#)



High-Temperature Synchrotron Powder Diffractometer

Wow!

Here are some highlights of recent XRDUC projects



Get in touch with User Center staff



Diffraction and Thermophysical Properties Group Home Page



High Temperature Materials Laboratory Home Page

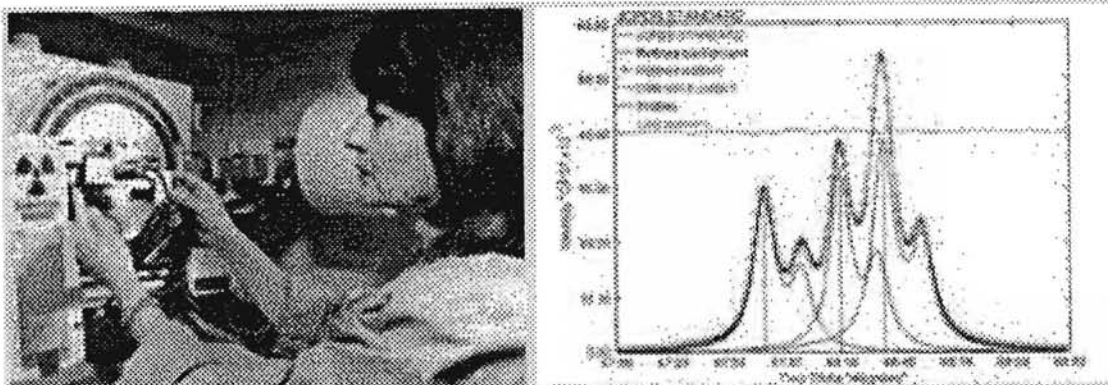


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Room Temperature X-Ray Diffractometer



Click on either picture to bring up a larger image

Features

- Scintag PADV Vertical Theta-2 Theta Goniometer
- Solid state detector - high signal to noise ratio
- Position-sensitive detector - 10x speed increase
- Choice of single or 4-position sample holder
- Choice of radiation (Cu, Mo, Cr, Co, Ag, Fe)

Applications

- Rietveld structure refinement
- Lattice parameter and solid solution composition determination
- Crystallite size and microstrain determination
- Quantitative analysis
- Phase identification




[Back to Diffraction User Center Home Page](#)



[Diffraction and Thermophysical Properties Group Home Page](#)



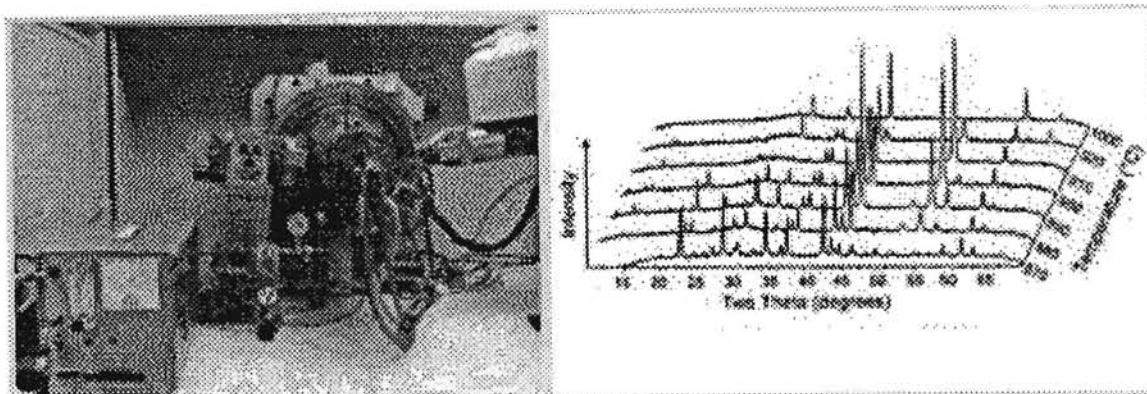
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High Temperature X-Ray Diffractometer



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Features

- Operates in oxidizing, reducing, or inert gas environments at temperatures up to 1600 C
- Operates in vacuum at temperatures up to 2700 C
- Solid-state germanium x-ray detector
- State-of-the-art data handling with peak search, deconvolution, automated pattern indexing, standard calibration, and lattice parameter refinement
- Complete computer automated search-match capability with entire Powder Diffraction File
- Rietveld and whole-pattern fitting capabilities for studies of structure and quantitative phase analysis
- Position sensitive detector for high precision measurements
- PO₂ control and sensor
- Operation possible in a pure hydrogen gas environment
- Choice of radiation (Cu, Mo, Cr, Co, Ag, Fe)

Applications

- Phase equilibria studies
- Order/disorder transformations
- Crystalline lattice thermal expansion calculation
- Measurement of oxidation kinetics
- In-situ process simulation



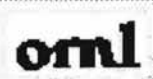
[Back to Diffraction User Center Home Page](#)



[Diffraction and Thermophysical Properties Group Home Page](#)



[High Temperature Materials Laboratory Home Page](#)

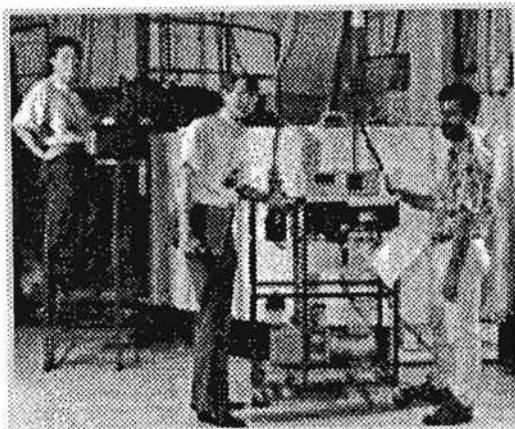


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High Temperature Neutron Powder Diffractometer



[Click to see a larger image](#)

Features

High Resolution 32 Detector Diffractometer

Structure refinement sensitive to light elements (H, C, O, S, etc)

Bulk probe - free from surface effects

High temperature furnace operates at temperatures from room temperature to 1600 C in vacuum

Cryostat operates at temperatures from 2 K to room temperature



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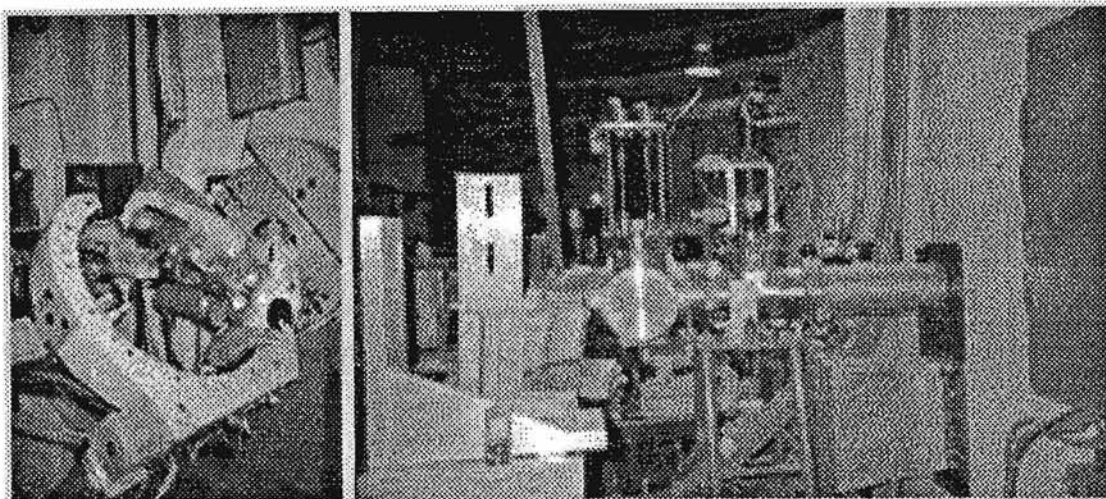


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High Temperature Synchrotron X-Ray Powder Diffractometer



Click on either picture to bring up a larger image

Facility

X14A Beamline at National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory

- ✦ Ultra-high resolution and intensity 6-circle powder diffractometer
- ✦ Focusing incident beam silicon monochromator
- ✦ Radiation wavelengths from 0.5 to 2.5 Angstroms
- ✦ High temperature furnace under construction

Measurement Capabilities

- ✦ Powder diffraction
- ✦ Residual stress
- ✦ Texture
- ✦ Grazing Incidence X-Ray Diffraction (GIXD)
- ✦ Extended X-Ray Absorption - Fine Structure (EXAFS)
- ✦ Single crystal diffraction



Click on either picture to bring up a larger image

Related Links

[National Synchrotron Light Source at Brookhaven National Laboratory
NSLS X14A Beamline](#)



[Back to Diffraction User Center Home Page](#)



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X-Ray Diffraction User Center Staff

All staff members may be reached by U.S. Mail at:

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[Back to Diffraction User Center Page](#)



[Diffraction and Thermophysical Properties Group Home Page](#)



[High Temperature Materials Laboratory Home Page](#)

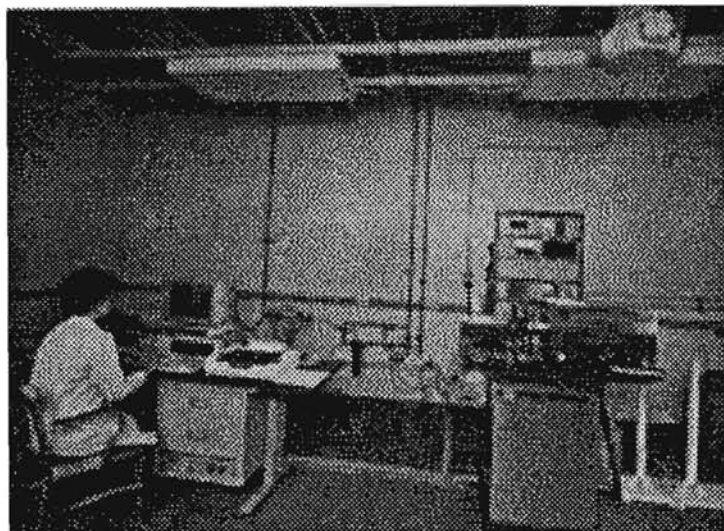


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PHYSICAL PROPERTIES USER CENTER



The Physical Properties User Center is used to study thermal stability, expansion, and thermal conductivity of materials to 1400 degrees C. An automated xenon-flash instrument allows diffusivity measurements to be made at room temperature. A thermal conductivity microprobe, a new class of instrument for determining heat flow in solid surfaces on a scale of a few micrometers, has just become operational. Other new instruments include a Laser Flash Thermal Diffusivity System and an infrared camera.

Instruments

Thermal Analysis:

- Simultaneous Thermal Analysis/Mass Spectrometer
- Differential Scanning Calorimeter
- Dual Push Rod Dilatometer
- Concurrent Thermal Analysis
- Pycnometer

Thermal Transport:

- High-Temperature Laser Flash Unit
- Room-Temperature Xenon Flash Unit
- Longitudinal Bar Thermal Conductivity Cryostat
- Scanning Thermal Conductivity Microscope

[Infrared Camera](#)

[3-Omega](#)

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[Highlights of recent Physical Properties User Center projects](#)



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Simultaneous Thermal Analysis



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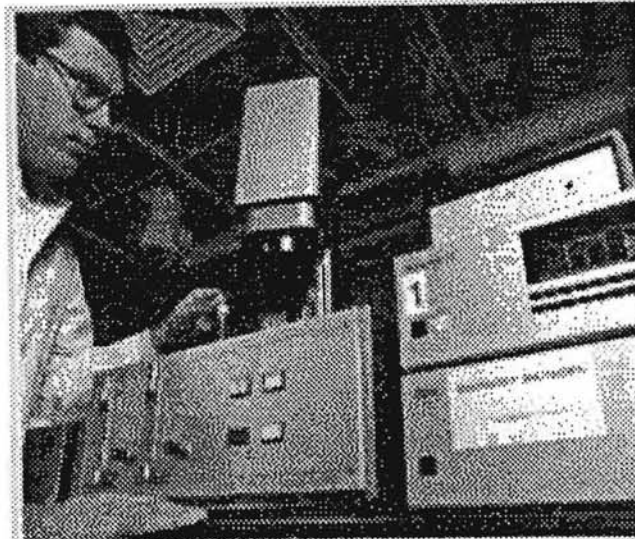
Simultaneous Thermal Analysis (STA) techniques comprise both differential thermal analysis (DTA) and thermogravimetry (TG). A mass spectrometer (MS) is attached to the STA instrument for evolved gas analysis. STA operates in a manner similar to differential scanning calorimetry (DSC). Two sample crucibles are heated or cooled at a precisely controlled rate in a controlled environment. One crucible contains a standard of known thermal response; the unknown is placed in the second crucible. Differences in the thermal behavior of the two materials caused by differences in specific heat, occurrence of an exothermic or endothermic reaction, or a phase change result in a temperature difference between the two crucibles.

Temperature differences, measured with a Pt vs. Pt-10Rh differential thermocouple, permit properties of the unknown to be determined relative to that of the standard. Simultaneously, any change in mass of the unknown sample during a heating cycle can be measured with a microbalance as a function of temperature. During the thermal cycle, a controlled leak allows samples of the gaseous environment in the immediate vicinity of the specimen to be drawn into a quadrupole mass analyzer. Evolved gases can thus be identified by the mass-charge ratios of molecules or atoms in the gas.

Features

- DTA and TGA measurements from 25 to 1450 C

Differential Scanning Calorimeter



[Click on picture to see a larger image](#)

The operation of a differential scanning calorimeter (DSC) is based on measurement of the thermal response of an unknown specimen as compared with a standard when the two are heated uniformly at a constant rate. The Stanton-Redcroft DSC consists of a furnace containing two identical crucibles, each of which rests on a thin plate located inside the measurement head. Directly beneath the center of each crucible is the junction of a Pt vs. Pt-10Rh differential thermocouple. Any difference in temperature of the two specimens is caused by differences in mass, specific heat, heats of reaction, or phase transitions.

To determine specific heat capacity, a baseline is established by measuring the temperature differential of the empty crucibles as the temperature is changed at a constant rate over the temperature range of interest. Thermal response records are then acquired for a standard material (usually sapphire) and an unknown under identical conditions. The ratio of the departure of the standard and unknown from the baseline is then used to calculate the specific heat of the unknown.

Features

- Operation of instrument completely automated and computer controlled
- Operation in oxygen, air, or inert gas environments to 1450 C
- Specific heat measurements to about 1200 C with +/- 3.5% error
- Small sample volumes required (0.2 to 20 cubic mm)

– Most rapid method for determining specific heat capacity

Applications

The DSC is used to measure specific heat capacity (100 to 1000 C) and heats of transition, and to detect phase changes and melting points in the range of 20 to 1450 C. Specific heat capacity can be used in conjunction with thermal diffusivity to obtain thermal conductivity.



[Back to Physical Properties User Center Page](#)



[Diffraction and Thermophysical Properties Group Home Page](#)



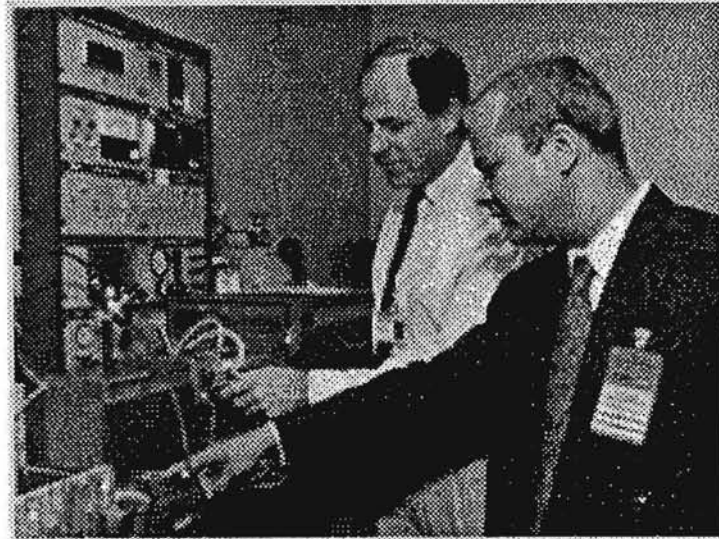
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Dual Push-Rod Dilatometer



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The Physical Properties User Center is equipped with a differential push rod dilatometer which measures linear thermal expansion (LTE) at temperatures up to 1500 C. The thermal expansion of a test specimen is determined relative to that of a standard reference specimen. The two samples are placed side by side in a furnace and two alumina push rods that extend from the furnace to a thermally isolated linearly variable displacement transducer (LVDT) bear on the samples. The difference in expansion between the two specimens results in the differential movement of the push rods, thus allowing the linear thermal expansion of the unknown sample to be determined. The temperature of the specimen is measured with a Pt vs. Pt-10Rh thermocouple to a resolution of 0.1 degrees C.

Features

- Measurements from 20 to 1500 C
- Differential length changes measured to +/- 1.5%
- Measurements in both inert gas and vacuum environments
- Dedicated computer control of time-temperature programs for the samples
- Length-change data automatically acquired, analyzed, and plotted

Applications

In addition to measuring a material's coefficient of thermal expansion as a function of

temperature, the dilatometer can serve as a probe to detect various physical or chemical changes in a specimen. For example, the occurrence of a phase change or the approach to the Curie point of a sample is almost always accompanied by a change in thermal expansion. The dilatometer can also be used to follow the course of a sintering process.



[Back to Physical Properties User Center Page](#)



[Diffraction and Thermophysical Properties Group Home Page](#)



[High Temperature Materials Laboratory Home Page](#)



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Concurrent Thermal Analysis

Concurrent thermal analysis comprises both thermogravimetry (TG) and differential thermal analysis (DTA). Separate TG and DTA units are used, thus allowing optimization of the size and shape of each sample. Large samples can be used for TG, while smaller samples are used in DTA. Analysis of the samples is made concurrent by using one temperature controller to closely match the temperature of the two samples as they are heated or cooled at a precisely programmed rate. TG analysis can be run in controlled environments including high-vacuum $5 \times 10E-6$ torr, inert gases, and corrosive gases. With use of an inert gas, the exit gas stream from the TG unit can be directed through the DTA unit if desired.

Features

- Concurrent TG and DTA measurements from 20 to 1700 C
- TG balance capacity up to 100 g with sample diameters up to 30 mm
- Mass change determinations to +/- 0.01 mg
- Heating rates up to 100 C /min
- Controlled environments including vacuum, inert, and corrosive gases
- All systems fully computer controlled

Applications

Concurrent thermal analysis can be used to examine the time temperature-environment relationships for high temperature materials. The ability to accommodate large samples allows the testing of real components in aggressive environments simulating actual processing or operating conditions.



[Back to Physical Properties User Center Page](#)



[Diffraction and Thermophysical Properties Group Home Page](#)



[High Temperature Materials Laboratory Home Page](#)



[Oak Ridge National Laboratory Home Page](#)

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Auto-Pycnometer for Solid Density Measurements

The Penta-Pycnometer is capable of measuring the true density of solid materials. Up to five samples can be loaded and purged simultaneously. Each of the five samples is then analyzed automatically in sequence with no operator involvement. Each sample can be analyzed automatically as many times as is required until a user-selected reproducibility is obtained or until a user-selected maximum number of runs is completed. The unit also features automatic calibration and automatic transducer zero reset prior to each run. Front panel LEDs display the operational status, and different sample cell sizes can accommodate a wide variety of samples.

Features

- Sample volumes from 0.1 to 135 cubic centimeters
- Sample cell volumes of 4.5, 10, 50, and 135 cubic centimeters
- Accuracy better than +/- 0.03%
- Reproducibility better than +/- 0.015%
- Self-calibrating, with calibration values permanently maintained in memory until recalibration

Applications

Pycnometers are currently used for research and quality control in such diverse industries as ceramics, catalysts, fillers, nuclear fuels, petrochemicals, carbon blacks, charcoals, fibers, minerals, cement, powdered metals, resins, and many others.



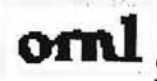
[Back to Physical Properties User Center Page](#)



[Diffraction and Thermophysical Properties Group Home Page](#)



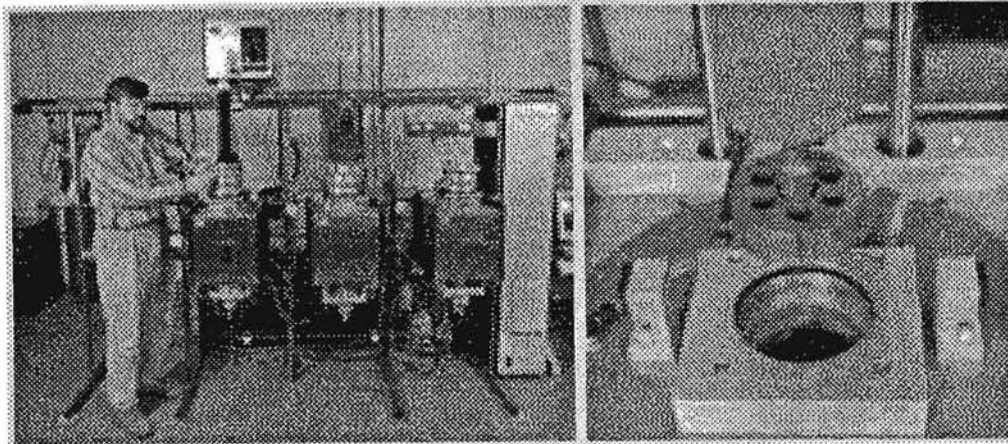
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Laser Flash Thermal Diffusivity System



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The laser-flash thermal diffusivity system is designed to acquire data automatically, from cryogenic temperatures up to 2500 C. It is used in the study of factors affecting the thermal transport properties of materials. A small, disk-shaped specimen, 12.4 mm in diameter and 1 to 8 mm thick, is placed in an evacuated tube furnace and oriented with its flat surfaces perpendicular to the furnace axis. A neodymium/glass laser is used to supply a high-intensity, short-duration pulse of thermal energy to one face of the test specimen. The intensity of the beam is controlled by varying the laser power supply and use of attenuating filters. The resulting temperature rise of the other face of the test specimen is monitored as a function of time by an indium antimonide infrared (IR) detector and stored in computer memory. The thermal diffusivity is determined from a numerical analysis of the IR detector output. The computer also controls all vacuum operations, amplifier settings, scan times, and furnace temperatures. Users typically learn the menu-driven computer software in less than one working day.

Features:

- Extended temperature range (-150 to 2500 C)
- Focused quartz lamp furnace for quench/shock studies
- Dilatometer attachment
 - High temperature (2500 C)
 - Bi-layer (1450 C)
- Accepts molten metals (to 1700 C), solids, and powders
- Measures multilayer samples

Enhanced environmental options (vacuum, inert, oxidizing, or reducing atmospheres)

Measures electrical resistivity of metals

Rapid numerical data analysis of temperature rise vs. time

Finite pulse width and heat-loss corrections

Two- and three-layer systems and thermal contact resistance measurements

Step-heat-flux thermal diffusivity measurements

Complete computer control of operations

Flexibility

• Six-sample carousel

• Unused electrical feed-thrus

• Other thermal property measurements

Applications

Thermal conductivity is often a property of great interest in the development of high-performance materials. However, thermal conductivity measurements at high temperatures are difficult and time consuming, and they require relatively large specimens. Thermal conductivity may be calculated from measurements of thermal diffusivity, specific heat, and bulk density. This method for obtaining thermal conductivity is relatively fast and requires only a small amount of material, an important consideration in research on new, experimental materials. The LFTD technique has been used extensively in the study of factors affecting the properties of materials, such as processing conditions, composition, heat treatments, sintering aids and dopants, composite structure, grain size, and porosity. The technique has been applied to a wide range of materials including glasses, plastics, metals, ceramics, composites, crystals, and foams.



[Back to Physical Properties User Center Page](#)



[Diffraction and Thermophysical Properties Group Home Page](#)



[High Temperature Materials Laboratory Home Page](#)



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Xenon Flash Thermal Diffusivity System



Reliable measurement of thermal diffusivity can in many cases be obtained through the flash technique. In this method, a short pulse (less than 1 millisecond) of heat is applied to the front face of a specimen using a xenon flash lamp, and the temperature change of the rear face is measured with an infrared (IR) detector. The xenon flash thermal diffusivity system is optimized for room-temperature measurements. The flash method, however, may not be applicable to heterogeneous materials when the various phases do not thermally couple with each other. The step-heat-flux method is an alternative to the flash method and greatly increases the variety of materials that can be measured. Rather than a pulse of heat, the step-heat method applies a constant heat source to the front face of the specimen. The heat source is a 250-W halogen lamp. The rear face of the specimen is monitored by an IR detector, just as in the flash method; however, the resulting temperature curve is now linear with respect to time. Analysis of the curve gives thermal diffusivity as a function of time. The apparent change in diffusivity results from heat loss and is linear with respect to time. Extrapolating these data to time zero gives the thermal diffusivity value with zero heat loss.

Features

Optimized for room-temperature thermal diffusivity measurements of ceramics, metals, composites, and coatings.

- Rapid numerical data analysis of temperature rise vs. time.
- Finite pulse-width and heat-loss corrections.
- Accepts a wide range of specimen sizes and shapes.
- Complete computer control of operations.

Applications

The xenon flash and step-heat-flux thermal diffusivity system has been used in the study of factors affecting the properties of materials, such as processing conditions, composition, heat treatments, sintering aids and dopants, composite structure, grain size, and porosity. The technique is applicable to a wide range of materials including glasses, plastics, metals, ceramics, composites, crystals, and foams. The system can measure the same specimen prepared for the laser flash system as well as other geometries, including plates, cubes, bars, and irregular shapes. The thermal diffusivity of coatings can also be measured when the density, thickness, and specific heat of both the coating and the substrate are known. The effective thermal diffusivity of laminar composites can be measured by using the step-heat-flux technique.



[Back to Physical Properties User Center Page](#)



[Diffraction and Thermophysical Properties Group Home Page](#)



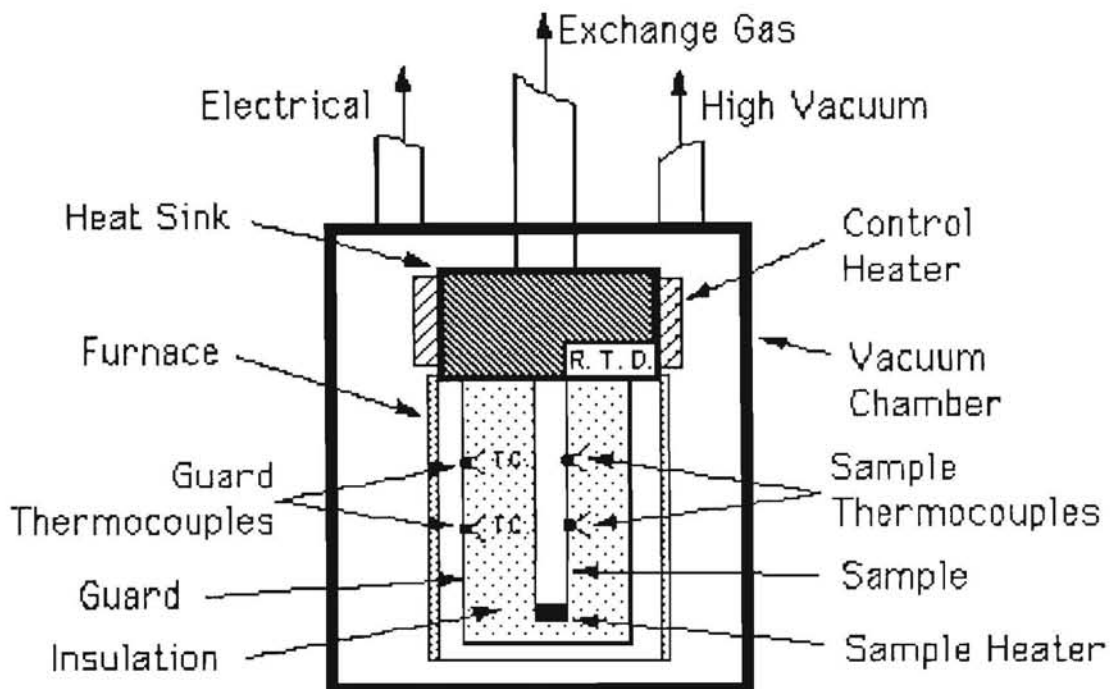
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Guarded Longitudinal Bar Thermal Conductivity Cryostat



The Guarded Longitudinal Bar Cryostat represents a steady-state technique to directly measure thermal conductivity. The instrument is designed to operate from cryogenic temperatures up to 200 C under high vacuum. The bar-shaped test specimen is mounted to a temperature controlled heat sink. A small heater is applied to the opposite end of the specimen and produces a temperature gradient along the specimen. This temperature gradient is measured by two differential thermocouples. To minimize radiation heat loss from the test specimen, it is surrounded by a guard with a matching temperature gradient. Thermal conductivity is calculated from a knowledge of the temperature gradient of the specimen, the cross-sectional area of the specimen, and the power dissipated in the specimen heater. The vacuum system, temperature control and sequencing, and data acquisition and analysis are fully automated and controlled by menu-driven software. The cryostat can simultaneously measure electrical resistivity of metal specimens over the entire temperature range.

Features

- Measures thermal conductivity from cryogenic temperatures up to 200 C
- Requires relatively small bar- or rod-shaped specimens (typical flexure strength bars)
- Measures thin, high thermal conductivity specimens
- Automatic temperature sequencing and control
- Complete computer control of vacuum operations and data acquisition
- Easy-to-learn menu-driven software
- Option for measuring electrical resistivity

Applications

Low-temperature thermal conductivity measurements have been used extensively in the study of factors affecting the properties of materials, such as processing conditions, composition, heat treatments, sintering aids and dopants, composite structure, grain size, chemical impurities, lattice defects, and porosity. The technique has been applied to a wide range of materials including metals, ceramics, composites, and diamond films.



[Back to Physical Properties User Center Page](#)



[Diffraction and Thermophysical Properties Group Home Page](#)



[High Temperature Materials Laboratory Home Page](#)



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Scanning Thermal Conductivity Microscope

The scanning thermal conductivity microscope (STCM) is capable of producing two-dimensional thermal conductivity maps of specimen surfaces with sub-micron resolution. This instrument is based on an atomic force microscope with a probe modified to measure thermal conductivity. The temperature of the probe is elevated slightly above that of the test specimen. When the probe tip is brought into contact with the test specimen, the probe tip will cool. The amount by which the probe will cool is related to the thermal conductivity of the test specimen in the region of contact. The probe is located at the end of a long, thin cantilever, which is rastered across the surface of the test specimen by means of piezoelectric translators. Topographic information comes from the voltage applied to the Z-piezoelectric stack; simultaneously, thermal conductivity information comes from the thermometer located on the warmed probe. The topographic and conductivity information is associated with a position on the specimen surface through the voltages applied to the X- and Y-piezoelectric stacks. This provides a means of visually correlating thermal conductivity with microstructural features. The thermal conductivity probe is calibrated by using standard reference materials and high-purity materials.

Features

- Produces topographic and thermal conductivity images simultaneously
- Measures relative room-temperature thermal conductivity with sub micron resolution
- Maps functionally gradient coatings, intergranular phases, powders, fibers, thin films, metals, ceramics, glasses, superconductors, composites, etc.
- Complete computer control of data acquisition
- Easy-to-learn menu-driven data acquisition and image analysis software
- Short measurement time

Applications

Microscopic thermal conductivity measurements provide a better understanding of bulk thermal conductivity measurements and enable measurements of microscopic structures that are difficult or impossible to accurately characterize with traditional techniques. Simultaneous topographic and thermal conductivity images will allow visual correlation between thermal conductivity and microstructural features. The STC microscope can also measure the postprocessed constituent properties of composite materials. This furnishes valuable information on the effect that processing, environmental exposure, and thermomechanical history have on the thermal conductivity of materials.



[Back to Physical Properties User Center Page](#)



[Diffraction and Thermophysical Properties Group Home Page](#)



[High Temperature Materials Laboratory Home Page](#)

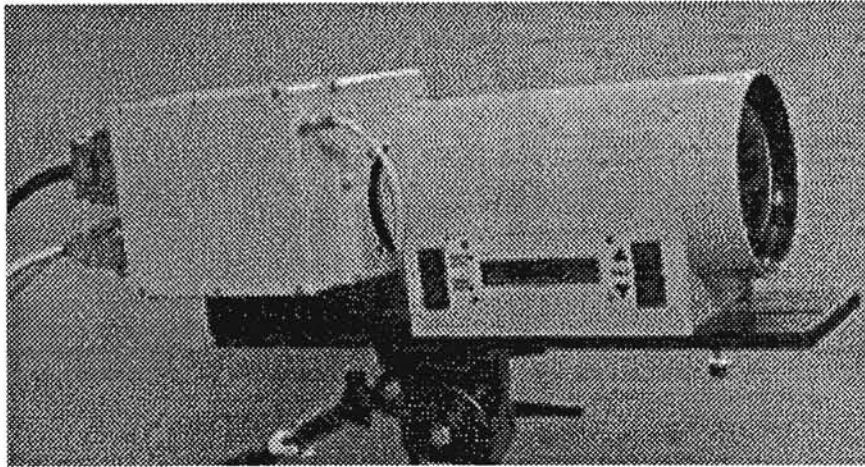


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High Speed Infrared Camera



Features

- Advanced image analysis software
 - Thermography
 - Thermal diffusivity
- 256 x 256 pixel resolution InSb Focal-plane array
- 120 to 1480 frames per second
- Temperature resolution greater than 0.015 C
- Field of view greater than 4 mm
- Motorized five-position filter wheel for high temperature studies
- Snap shot mode
- Closed-cycle sterling linear cooler
- Pulse and step heating

Applications

- Thermal diffusivity maps
- Detection of subsurface flaws



[Back to Physical Properties User Center Page](#)



[Diffraction and Thermophysical Properties Group Home Page](#)





High Temperature Materials Laboratory Home Page



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3-Omega Thermal Conductivity Measurement

Features

- Non-conductors
- Bulk samples
- Small samples
- Thin Films
- Temperature range of -190 to 500 C



[Back to Physical Properties User Center Page](#)



[Diffraction and Thermophysical Properties Group Home Page](#)



[High Temperature Materials Laboratory Home Page](#)



[Oak Ridge National Laboratory Home Page](#)

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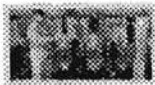
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[Back to Physical Properties User Center Page](#)



[Diffraction and Thermophysical Properties Group Home Page](#)



[High Temperature Materials Laboratory Home Page](#)



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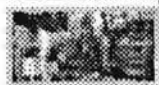
Residual Stress User Center

Residual stresses affect such important materials design properties as fatigue life, fracture strength, onset of yield, and microcracking. X-ray and neutron diffraction methods are available to measure macro- and micro-residual stresses in polycrystalline and single-crystal materials, as well as crystallographic texture.

Neutron diffraction facilities are complementary to the traditional x-ray diffraction facilities because of the great differences in a material's absorption of the two radiation types. Neutrons can penetrate many millimeters into most engineering materials, while x-rays are absorbed within a surface layer several micrometers thick. Thus, neutron diffraction can be used to probe strains throughout the volume of a material. X-rays are used to measure the strain in a shallow layer at the surface.



[X-Ray Measurements](#)



[Neutron Measurements](#)

Wow!

[Highlights](#) of recent Residual Stress User Center projects



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[High Temperature Materials Laboratory Home Page](#)

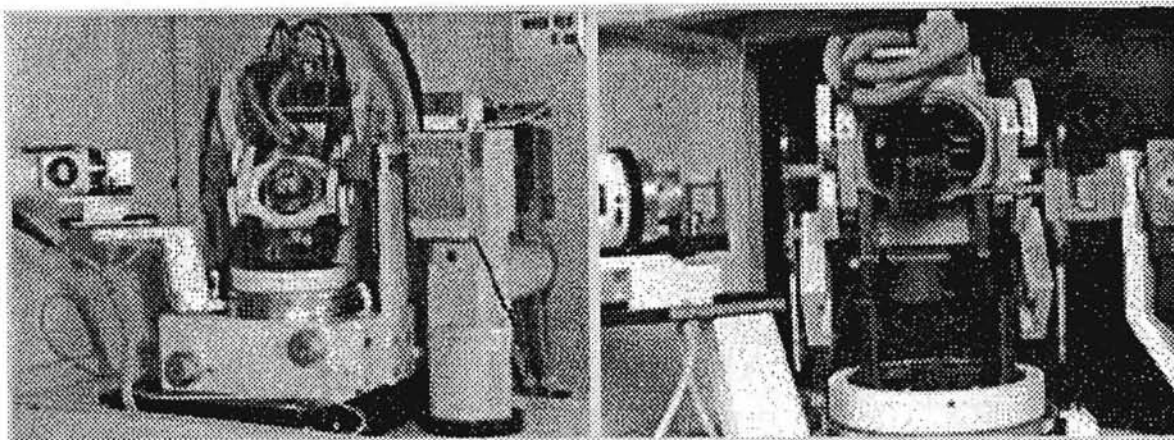


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RESIDUAL STRESS USER CENTER (X-RAY)



Instruments



4-Axis Goniometer/18-kW Rotating Anode X-ray Source



4-Axis Goniometer/2-kW X-ray Tube

Of all the residual stress measuring techniques, diffraction methods are widely accepted as the most general and reliable nondestructive method of quantifying the residual stress tensor. Diffraction methods use the atomic planes of the crystalline grains within the material as highly sensitive strain sensors. Analysis techniques permit separation of long range macrostresses and short range or grain-to-grain microstresses.

The Residual Stress User Center includes two X-ray diffractometers to measure residual stress and texture in and near the surface of ceramics and alloys. One Scintag PTS goniometer is equipped with an 18-kW rotating anode generator, while the second has a fixed target sealed tube X-ray source. Both systems provide a highly flexible sample tilt system, divergent or parallel beam optics, and Peltier-cooled detectors. A position-sensitive detector is also available. Sample masses of a few kilograms can be accommodated.

Applications

Macro residual stresses extend over a long range relative to the scale of the specimen's microstructure. These stresses originate from differential plastic flow, differential cooling rates, or phase transformations with volume changes. Macro residual stresses are created, for example, by welding, forging, casting, rolling, machining, surface treatments, or heat treating. These stresses are often high at the surface of a material, and their variation across a surface can be critical to material performance. Research areas include studies of the development of residual stress as a function of the rate and method of material removal or resulting from changes in a manufacturing process. Other studies include determination of residual stresses in surface treated materials for assessing the effectiveness of the surface treatment in introducing beneficial compressive stresses at the surface.

Microstresses typically originate from the differential thermal expansion between phases or differential plastic flow between grains. For example, microstresses that develop in composites primarily result from differences in the respective thermal expansion coefficients. Studies of ceramic composites include characterizing the stress as a function of fiber or whisker loading, coating, and processing, and can be determined at various temperatures.



[Back to Residual Stress User Center Page](#)



[Diffraction and Thermophysical Properties Group Home Page](#)



[High Temperature Materials Laboratory Home Page](#)

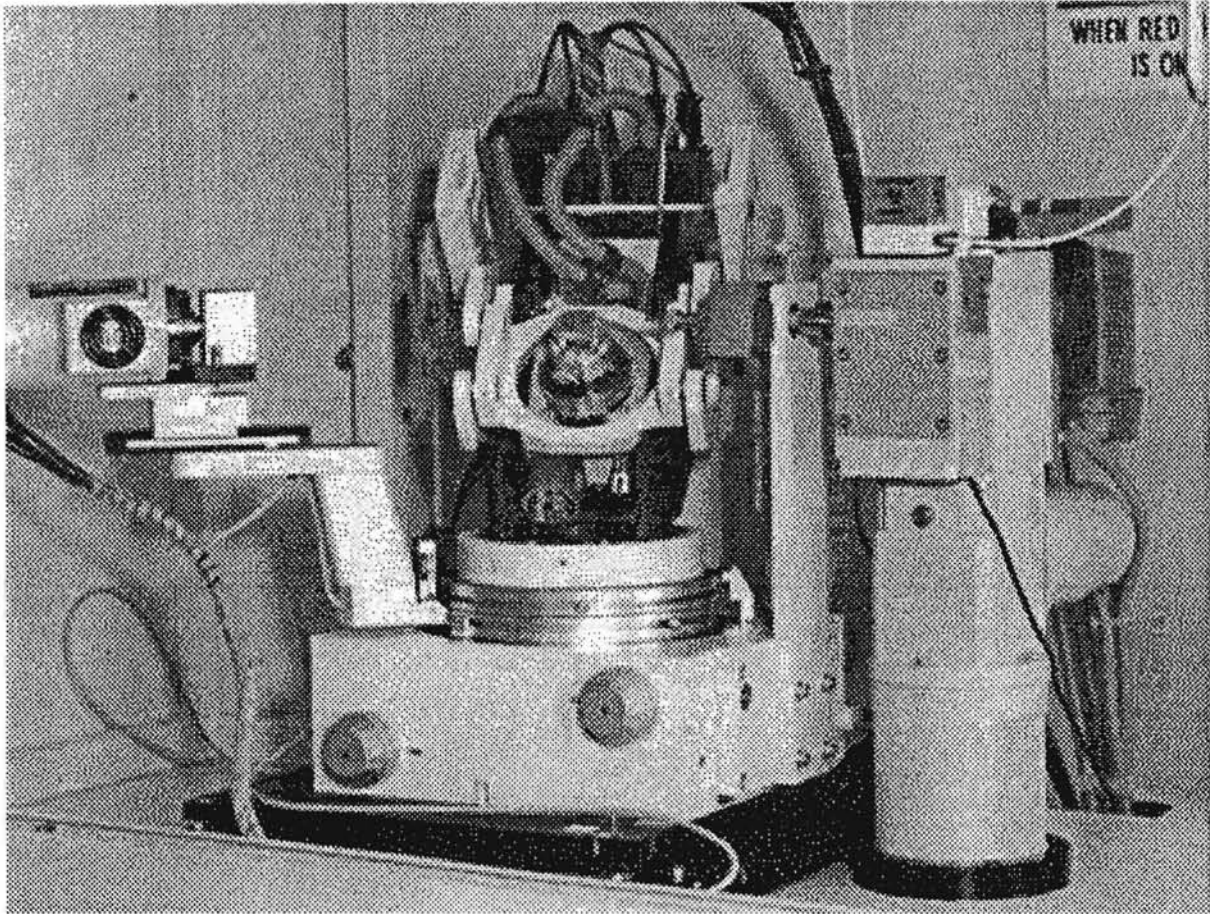


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4-Axis Goniometer with High-Flux Rotating Anode X-ray Source



The Scintag Polycrystalline-Texture-Stress (PTS) goniometer with high-flux rotating anode x-ray source is designed to permit mapping of the residual stress state across the surface of a specimen and to perform grazing incidence x-ray diffraction. This instrument permits measurement of either biaxial or triaxial residual strains in ceramics and alloys. It is also possible to determine elastic constants and to study the response of materials to applied loads.

Features

- High-flux, high-brilliance 18-kW rotating anode source with interchangeable anodes
- Divergent or near-parallel beam optics
- Unrestricted 2-theta range (from -20 to 162 degrees)
- Sample X and Y translations
- Highly automated, flexible data-collection options include psi or omega tilt along

with profile fitting of peak position

Solid-state detection of x-rays yields high peak-to-background ratios.

Optional position-sensitive detector available for rapid data collection

Specimen dimensions up to 140 mm in diameter, 40 mm in thickness, and 5 kg in mass

Software includes biaxial and triaxial stress analysis, pole figures, and orientation distribution function calculations from texture data

- Grazing incidence x-ray diffraction for depth profiling of stress and phase content
- Load cells for determination of diffraction elastic constants and furnaces for elevated temperature measurement of residual strain are being developed



[Back to X-Ray Residual Stress Page](#)



[Diffraction and Thermophysical Properties Group Home Page](#)



[High Temperature Materials Laboratory Home Page](#)

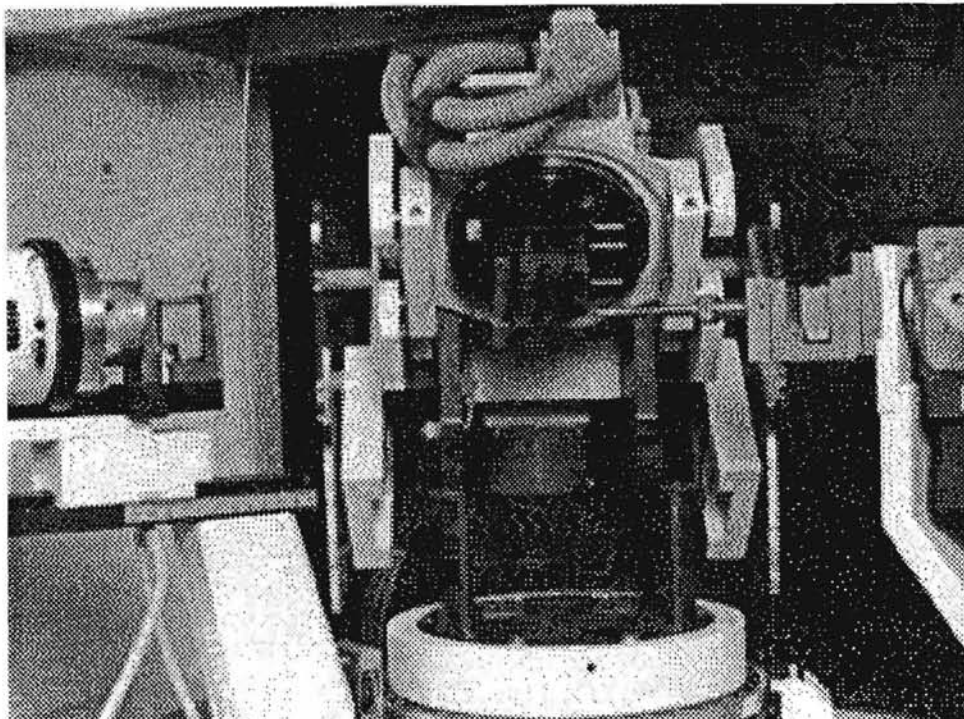


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4-Axis Goniometer with 2-kW X-Ray Tube



This Scintag Polycrystalline-Texture-Stress (PTS) goniometer, equipped with a sealed x-ray tube, can be used for residual stress measurements where small spot sizes are not required. It is also ideal for studies of crystallographic texture or preferred orientation by measuring one or more pole figures. Diffraction methods are widely accepted as the most general method of quantifying the texture of materials.

Properties of materials are heavily dependent on their texture, often a result of the manufacturing processes that include rolling, hot pressing, preferred crystalline growth, and epitaxy. Texture affects such important design and processing properties as yield, strength, corrosion resistance, formability, thermal transport, magnetic properties, and electrical conductivity.

Features

- Interchangeable, 2-kW x-ray tubes
- Divergent-beam and parallel-beam optics
- Scintag PTS four-axis goniometer for texture and stress analysis
- Unrestricted 2-theta range (from -20 to 165 degrees)

Sample X and Y translations

Solid-state detection of x-rays

- Specimen dimensions up to 140 mm in diameter, 40 mm in thickness, and 5 kg in mass
- Highly automated, flexible data collection and analysis options including multiple pole figure data collection and calculation of the orientation distribution function
- Stress analysis uses the same data collection and analysis routines as the high-flux rotating anode system



[Back to X-Ray Residual Stress page](#)



[Diffraction and Thermophysical Properties Home Page](#)



[High Temperature Materials Laboratory Home Page](#)



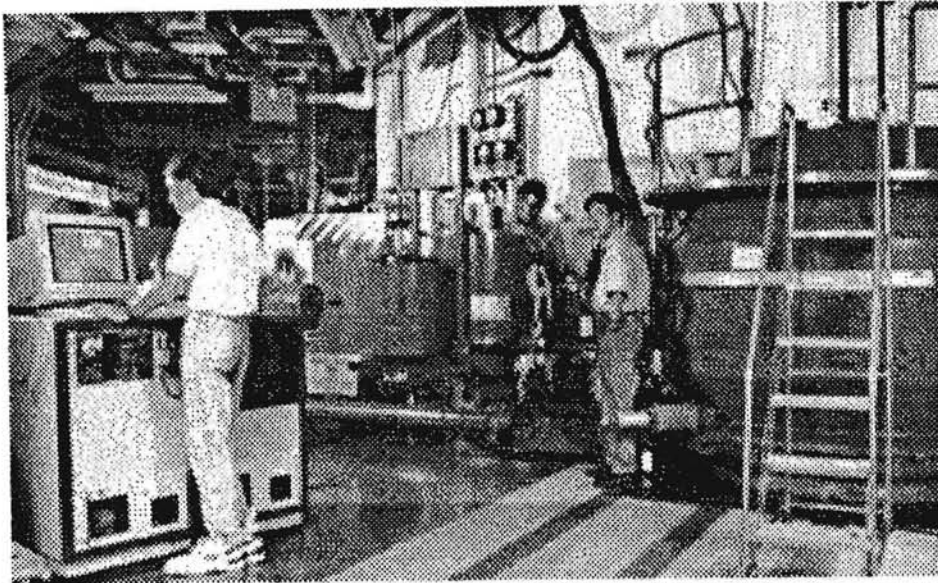
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RESIDUAL STRESS USER CENTER

(NEUTRON RESIDUAL STRESS FACILITY)



Neutron Diffraction Macro Residual Stress Mapping Facility Neutron Powder Diffraction Micro Residual Stress Facility

The Neutron Residual Stress Facilities were added in 1994 as an expansion of the capabilities of the Residual Stress User Center. The neutron diffraction facilities for residual stress measurement provide macro residual stress mapping and micro residual stress characterization.

The macro residual stress mapping facility currently is an attachment to a conventional triple axis spectrometer. Measurements of strains are possible over a large range of specimen sizes, e.g. from 6 mm diameter rods containing a functionally gradient joint to 600 mm (24 in) diameter steel cylinders with girth welds. Results may be used to quantify thermal stress relief effectiveness, to validate finite element models, and for failure analysis, design, and life prediction.

Design is underway for enhanced facilities which will include a special neutron spectrometer for rapid data collection plus new computer capabilities for data analysis. The triple-axis spectrometer instrumentation is located at the High Flux Isotope Reactor at ORNL. This facility, when equipped with the new spectrometer, will provide the capability to more rapidly measure and map the stress fields inside relatively large solid objects with

lateral dimensions of a few feet.

The micro residual stress facility currently utilizes the high resolution neutron powder diffractometer managed by the Neutron Scattering Group, Solid State Division, ORNL. Time is available for user projects on this spectrometer until the new spectrometer is completed.

Related Links:

[High Flux Isotope Reactor \(HFIR\) at ORNL
Neutron Scattering Group, Solid State Division, ORNL](#)



[Back to Residual Stress User Center Page](#)



[Diffraction and Thermophysical Properties Group Home Page](#)



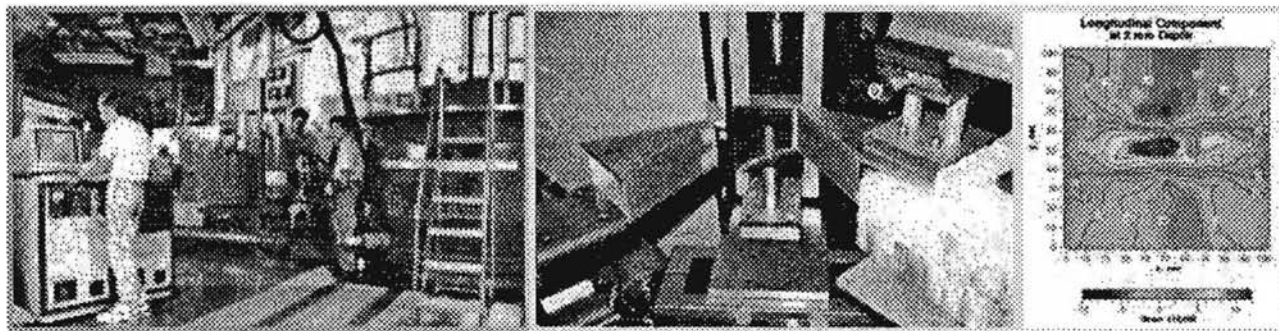
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Neutron Diffraction Macro Residual Stress Mapping Facility



Click on any of the above pictures to bring up a larger image

The High Flux Isotope Reactor at ORNL produces a beam of thermal neutrons from which a single-wavelength beam is selected by a crystal monochromator. The particular monochromator crystal and diffraction angle are selected to locate a particular diffraction peak at around 90 degrees 2-theta. The strain is determined by measuring the Bragg diffraction peak position and comparing it with the position from a strain-free sample.

A high resolution linear position sensitive detector is used to simultaneously record the full diffraction peak profile. Incident and diffracted beam slits are used to define the sampling or gage volume within a specimen. Measurements have been successfully completed using gage volumes as small as one cubic millimeter in materials such as iron and nickel. Enhanced instrumentation under development should improve this by a factor of 10.

Features:

Specimen-positioning equipment:

- X, Y, and Z translations of +/- 100 mm, with precision of +/- 0.02 mm
- Specimen and detector rotations
- Specimens to 100 kg and 40 cm x 40cm x 20cm

Highest-flux thermal neutron source in the United States

Selectable wavelength and diffraction angles (Be <11.0> monochromator)

ORDELA Position sensitive detectors:

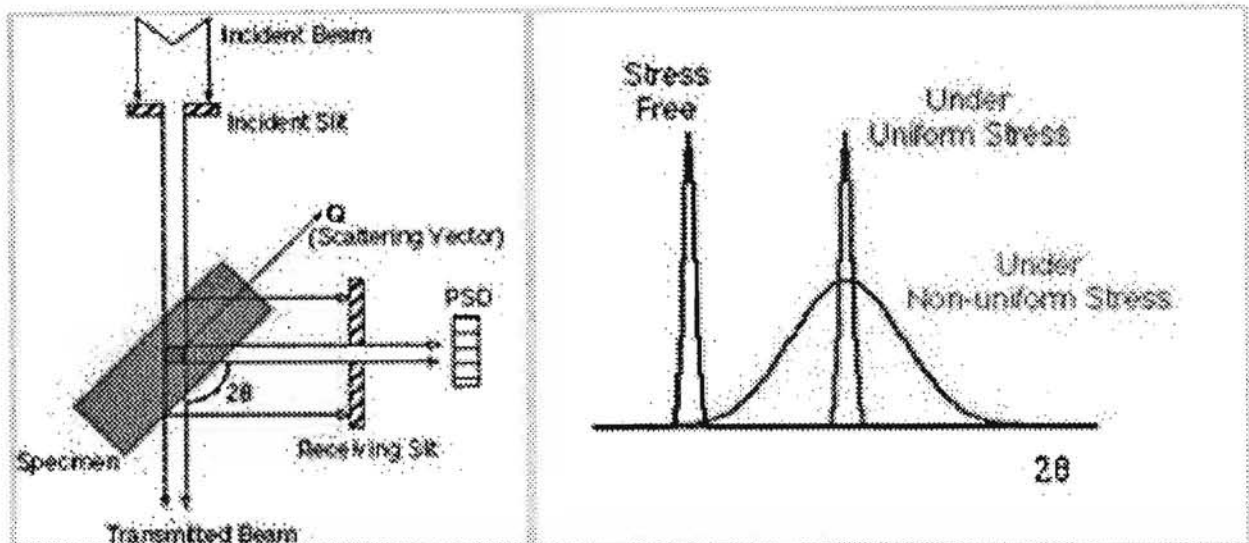
- Active area of 2.5 by 10 cm
- Seven-detector array in development

Automated data collection and on-line analysis

- Multilocation automated data collection
- Position-sensitive detector and scattering-angle calibration
- Peak profile fitting

- Gage volume of 1 to 20 cubic mm, depending on scattering power of material and required resolution
- Typical peak position fitting precision to ± 0.003 degrees 2-theta
- Triaxial strain measurement and stress analysis
- Load frame for tensile loading:
 - Diffraction elastic constants
 - Multiphase response to load in composites

How It Is Done



Click on either picture to bring up a larger image



[Back to Neutron Residual Stress Facility Page](#)



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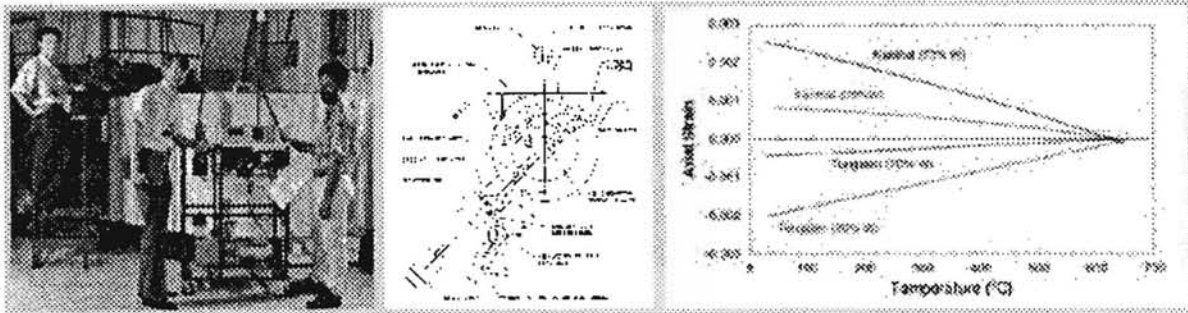
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Micro Residual Stress Using the Neutron Powder Diffraction Facility



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Micro residual stresses typically result from the difference between the thermal expansion of the different phases in a composite or other multiphase material. After cooling from the processing temperature, the thermal expansion mismatch can lead to large residual microstresses being created between the grains. The magnitude of these stresses can contribute to the toughening of a composite or lead to microcracking upon thermal cycling. Measurement of the micro residual stress state as a function of temperature can provide (1) a direct determination of the bond strength between phases in a composite, (2) a measure of the degree of microcracking, and (3) the temperature at which each phase is stress free.

Because thermal neutrons are highly penetrating, they are used to measure the specimen volume average microstresses, free from any surface effects. The full diffraction pattern is measured over a few hours and used to analyze the microstresses. Rietveld pattern refinement techniques are typically used to fit the lattice parameters and volume fraction of the constituent phases for each sample and the strain free constituents. Analysis of the shifts in lattice constants yields the strains.

Features:

- Monochromatic beam
- Thirty-two Helium-3 detectors with soller slits
- Pattern from 10 to 130 degrees 2-theta
- High resolution
- Non-ambient capabilities
 - High-temperature furnace
 - Low-temperature cryostat
- Automated data collection
- Rietveld analysis codes

☞ [Powder diffraction analysis codes](#)



[Back to Neutron Residual Stress Facility Page](#)



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[Back to Residual Stress User Center Page](#)





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