X-ray Tomographic Microscopy for Non-Destructive Inspection and Advanced Materials Characterization

Final Report
CRADA No. TC-0113-91

Date: 04/27/95

TACT:

Revision: 1

A. Parties

LLNL--96013989

The project is a relationship between the Lawrence Livermore National Laboratory (LLNL) and General Electric Aircraft Engines.

University of California
Lawrence Livermore National Laboratory
Attn. John Kinney
7000 East Avenue, L-356
Livermore, CA 94550

General Electric Aircraft Engines
Quality Technology Center
Attn. Mr. Ralph Isaacs
10270 Saint Rita Lane, Q-8
Cincinnati, Ohio 45215

B. Project Scope

This was a CRADA to transfer the x-ray tomographic microscope technology to the Quality Technology Center of General Electric Air Craft Engines Division in Ohio. The x-ray tomographic microscope (XTM) is a high resolution, three-dimensional imaging system that can be used to materials microstructures noninvasively. The apparatus consists of an x-ray source, x-ray collimators, sample positioning stages, a fluorescent screen to convert x-rays to visible light, an optical lens, and a thermoelectrically cooled charge coupled device detector. The details of the microscope's design have been described elsewhere. In practice, the sample to be imaged is positioned on a rotating stage. The sample is initially translated out of the x-ray path, and an image is obtained of the incident x-ray beam (the reference image). Next, the sample is placed between the x-ray path and the scintillator, and another image, the projection image, is acquired. The ratios of the logarithms of the reference image and the projection image provide values of the integrated x-ray attenuation through the sample. By rotating the sample in discrete angular increments through 180 degrees, enough data can be obtained to reconstruct the two-dimensional projection images into a three-dimensional image of the mineral density distribution in the sample. This reconstruction procedure, known as reconstruction from projections, is usually performed with the technique of Fourier-filtered back-projection. Using the present microscope, a 1 cubic centimeter volume can be imaged with five micrometer volume elements in about one hour or less.

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The ability to nondestructively image materials microstructures with the XTM, as opposed to imaging large scale flaws as is currently done, will enable GE to study complex problems in materials failure in a more statistically significant and cost effective manner. The application of XTM to advanced composites, where complex fiber architectures require the use of a three-dimensional imaging technique, will improve upon the existing models of failure analysis and will lead to more rapid development and application of new materials.

C. Technical

The project originally had the following milestones:

- a: Complete scoping of work defining specific GE needs and existing GE resources.
Establish guidelines for size and shielding requirements.
- b: Optics design completed and sent out to vendor.
- c: Completed system design.
- d: Optics fabricated and delivered to GE.
- e: Remaining hardware components finished.
- f: XTM system installed and operational at GE QTC Cincinnati.
- g: XTM certified, performance characteristics measured. System operational.

During the course of the CRADA, there were significant deviations from these original milestones caused by delays in processing the CRADA. As a result of the delays, LLNL would not participate in the assembly of the optical components, and GE would prepare the technical drawings for outside vending from design drawings provided by LLNL. This changed milestones b and c to one where LLNL designed the optical and general x-ray system and provided design drawings (see Attachment II) for GE to assemble into their own technical drawings. The CRADA eliminated milestone d, and significantly reduced the dollars award to LLNL.

The technology has now been successfully transferred to GE, and an operational system (now called the high-resolution volume CT, or HRVCT) is now in place. GE has taken several high resolution images of composite materials, and it is anticipated that HRVCT will provide a valuable new inspection capability at the Quality Technology Center.

D. Partner Contribution

General Electric provided x-ray equipment, a lead-shielded room, sample manipulators, x-ray tubes and controllers, a Photometrics CCD camera, all computers and interfaces, and software system modules required for acquiring and analyzing micro-CT images. In addition, GE provided engineering manpower and facilities support for the construction of the facility.

E. Financial (\$K)

Federal	\$500
Non-Federal	<u>317</u>
Total Funding	\$817

12-1-92 Review Date: TACT recommends financial increase of \$62K

COST PROFILE

Approved Funding
Profile (SK)

	FY92	FY93	FY94	FY95	FY96	Total
Planned Funding	300	138	62	0	0	500
Participant Cont.	26	158	132	0	0	317
WAs Issued	300	138	62	0	0	500

[illegible]

Attachment I contains documents that were part of the technology transfer project. These documents consist entirely of engineering drawings that were transferred to GE as part of the CRADA effort. The primary set of drawings is the XTM (x-ray tomographic microscope) as developed by LLNL. The second series of drawings are the modifications to the original design requested by GE. GE has subsequently changed the scintillator package housing as mentioned above. LLNL does not have, and does not need, copies of these design changes.

No significant properties were acquired by LLNL during this project. No proprietary information was exchanged with GE.

- 1) The Participant has reviewed the final report and concurs with the statements made therein.
- 2) The Participant agrees that any modifications or changes from the initial proposal were discussed and agreed to during the term of the project.
- 3) The Participant certifies that all reports, inventions disclosures and patent applications attributable to the project are completed or are in process and a list is attached to this report.

Lloyd L. Chase 4/27/95
Lloyd L. Chase Date
LLNL Project Program Mgr

Attachment I – Accomplishments Summary
Attachment II - Design Drawings

CRADA Title

Project Accomplishments Summary (Attachment I) CRADA No. TC-0113-91

Date: 04/24/95

Revision: 1

A. Parties

The project is a relationship between:

University of California
Lawrence Livermore National Laboratory
Attn. John Kinney
7000 East Avenue, L-356
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General Electric Aircraft Engines
Quality Technology Center
Attn. Ralph Isaacs
10270 Saint Rita Lane, Q-8
Cincinnati, Ohio 45215

B. Background

Prior to the development of the x-ray tomographic microscope (XTM), nondestructive x-ray inspection techniques were applied for flaw detection, but lacked the spatial resolution necessary to characterize material microstructures. The XTM offered better than a thousand-fold increase in volume resolution, and allows researchers to characterize damage nucleation and growth in advanced composite materials. The technology complemented the strong capabilities in x-ray CT inspection at the Quality Technology Center at GE Aircraft Engines, where state-of-the-art inspection technologies are required to insure the safety and reliability of civilian and defense aircraft engines.

C. Description

This was a project to transfer the x-ray tomographic microscope developed under DOE Defense Programs at LLNL to the Quality Technology Center at GE Aircraft Engines in Cincinnati, OH. Lawrence Livermore National Laboratory (LLNL) leads in the development of a new form of x-ray microscopy known as X-ray Tomographic Microscopy (XTM). XTM allows non-invasive, three-dimensional visualization of materials microstructures with extremely high spatial resolution, and augments more traditional characterization techniques such as electron microscopy.

The ability to nondestructively image materials microstructures, as opposed to imaging large scale flaws as is currently done, will enable General Electric to study complex problems in materials failure in a more statistically significant and cost effective manner. The application of XTM to advanced composites, where complex fiber architectures require the use of a three-dimensional imaging technique, will improve upon the existing models of failure analysis and will lead to more rapid development and application of new materials.

D. Expected Economic Impact

The XTM has now been implemented as the High Resolution Volume CT (HRVCT) system at GE Aircraft Engines. The HRVCT system will allow GE to explore the application of new materials for aircraft engines. By establishing a better understanding of the microstructure and failure criteria in these new materials, it will be possible to speed up there application in new engine designs. The use of these materials, coupled with better inspection methods, will provide US engine manufacturers with a competitive advantage over their foreign competitors.

E. Benefits to DOE

The CRADA between GE and LLNL provided funding that helped keep the XTM technology viable during the immediate period between the ending of the x-ray laser project and establishment of more significant support from the National Institutes of Health and DOE's Fossil Fuels. It is now apparent that the XTM technology will provide a vital characterization service for the National Ignition Facility. With the nondestructive, microscopic imaging capability provided with the XTM, it will be possible to correct target processing flaws and to run more quantitative experiments on the NIF facility.

F. Industry Area

The aircraft industry is the principal beneficiary of the CRADA between LLNL and GE. However, other industry segments may see even greater impact. For example, the technology has now been expanded to include in vivo imaging of bone, and provides a unique opportunity to explore degenerative bone diseases such as osteoporosis. Major US pharmaceutical industries have either provided capital, or have expressed interest in, using the XTM technology to explore countermeasures for osteoporosis. In addition, oil producing companies have expressed interest in using the XTM technology to evaluate porosity in oil bearing rock or in monitoring catalyst degradation in oil refining.

G. LLNL Point of Contact for Project Information

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H. Company Size and Point(s) of Contact

General Electric Aircraft Engines Division employs about 20,000 people in the Cincinnati area. GE is one of two jet engine manufacturing companies in the US, with their engines seeing use on both civilian and military airframes. Points of contact include:

Ralph Isaacs	(513) 552-4727
Tom Birdwell	(513) 552-4754
Ben Nightingale	(513) 552-4645

I. Project Examples

Most of the project examples have been reported in the scientific literature. Additional information can be requested from the principal investigator, Dr. John H. Kinney, at Lawrence Livermore National Laboratory.

Publications (selected):

J.H. Kinney, D.L. Haupt, J.M. White, W.L. Bell, M. Balooch, S.J. Marshall, G.W. Marshall, Jr., "The Effects of Nd and Ho:YAG Laser-Induced Surface Modification on Demineralization of Dentin Surfaces", *J. Dent. Res* (submitted).

J.H. Kinney, M Balooch, D.L. Haupt, S.J. Marshall, and G.W. Marshall, "Mineral Distribution and Dimensional Changes in Human Dentin during Demineralization", *Journal of Dental Research* (in press).

J.H. Kinney, C.P. Henry, D.L. Haupt, and T.L. Starr, "The topology of percolating porosity in woven fiber ceramic matrix composites", *Applied Composite Materials* (in press).

J.H. Kinney, N.E. Lane, and D.L. Haupt, "In vivo, 3D morphometry of trabecular bone", *J Bone Miner Res* 10:264-272 (1995).

N.E. Lane, J.M. Thompson, D. Haupt, and J.H. Kinney, "Intermittent therapy with parathyroid hormone (PTH) increases trabecular bone mass but not connectivity in osteopenic rats", 41st Annual Mtg., Orthopaedic Research Society, (accepted, podium presentation).

A.A. Gies, S. Majumdar, J.H. Kinney, and J.C. Lotz, "Mean intercept length from power spectrum related to mechanical test", 41st Annual Mtg., Orthopaedic Research Society, (accepted).

J.H. Kinney, G.W. Marshall, and S.J. Marshall, "Three-dimensional mapping of mineral densities in carious dentin: theory and method", *Scanning Microscopy* 8:197-205 (1994).

J.H. Kinney, D.L. Haupt, M.C. Nichols, et al., "Three-dimensional perspectives of evolving microstructures". *Nucl Instr. Meth. A* 347:480-486 (1994).

T.M. Breunig, M.C. Nichols, J.S. Gruver, J.H. Kinney, and D.L. Haupt, "A servo-mechanical load frame for in situ, noninvasive imaging of damage development". *Ceramic Engineering and Science Proc.*, 15:410-417 (1994).

J.H. Kinney, N.E. Lane, S Majumdar, et al., "Three-dimensional, in vivo microscopy of trabecular bone architecture", *Transactions Orthopaedic Research Society*, 19:56 (1994).

T.M. Breunig, M.C. Nichols, J.H. Kinney, and D.L. Haupt, "In situ Three-Dimensional Characterization using Noninvasive X-ray Tomographic Microscopy", in *The first International Conference on Composites Engineering*, D. Hui, ed., pp 63-64 (1994).

J.H. Kinney, T.M. Breunig, T.L. Starr, et al., "X-ray tomographic study of chemical vapor infiltration processing of ceramic composites", *Science*, 260:789-792 1993.

E. Zywickz, J.H. Kinney, M.L. Sattler, T.M. Breunig, and M.C. Nichols, "Heterogeneous fibre microstructures and their influence on failure", *J. Microscopy*, 169:247-253 (1993).

T.M. Breunig, M.C. Nichols, J.H. Kinney, and D.L. Haupt, "Quantitative Microstructural Characterization of a Composite Material Using Destructive and Noninvasive Techniques", Ceramic Engineering and Science Proceedings, 14:500-509 (1993).

J.H. Kinney, D.L. Haupt, T.M. Breunig, M.C. Nichols, T.L. Starr, and S.R. Stock, "Time-evolution of Microstructure During Forced Flow Chemical Vapor Infiltration of a Continuous Fiber Ceramic Matrix composite", Ceramic Engineering and Science Proceedings, 14:1028-1037 (1993).

J.H. Kinney and M.C. Nichols, "X-ray Tomographic Microscopy using Synchrotron Radiation", Annu. Rev. Mater. Sci, (1992) 22:121-52

J. Release of Information

RELEASE OF INFORMATION

I have reviewed the attached Project Accomplishment Summary prepared by Lawrence Livermore National Laboratory and agree that the information about our CRADA may be released for external distribution.

Ralph Isaacs
Name Ralph Isaacs
Organization GEA
Title Staff Scientist

10-24-95
Date

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D. 14 and 11/17/152