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LASER LIGHT SCATTERING INSTRUMENT

Advanced Technology Development
Final Report
September 1993

Executive Summary

The objective of this Advanced Technology Development (ATD) project has been to provide sturdy, miniaturized laser light scattering (LLS) instrumentation for use in microgravity experiments. To do this, we assessed user requirements, explored the capabilities of existing and prospective laser light scattering hardware, and both coordinated and participated in the hardware and software advances needed for a flight hardware instrument. We have successfully breadboarded and evaluated an engineering version of a single-angle glovebox instrument which uses solid state detectors and lasers, along with fiber optics and for beam delivery and detection. Additionally, we have provided the specifications and written verification procedures necessary for procuring a miniature multi-angle LLS instrument which will be used by the flight hardware project which resulted from this work and from this project's interaction with the laser light scattering community.

Task Description

This Advanced Technology Development (ATD) project has provided a Laser Light Scattering (LLS) Instrument for use in microgravity experiments. To this end, we assessed user requirements, explored the capabilities of existing and prospective laser light scattering hardware, and both coordinated and participated in the hardware and software advances needed for a flight hardware instrument. We have breadboarded and evaluated a single-angle glovebox instrument, and we have provided the specifications and written verification procedures necessary for procuring a miniature multi-angle LLS instrument. The laser light scattering ATD effort transitioned to a flight hardware development project once the evaluations of the LLS proposals from several NASA Research Announcements were announced (for example, NRA 91-OSSA-17). The procurement and evaluation of proposals for a multi-angle prototype instrument has been transferred to the laser light scattering flight hardware project.

We chose a modular approach for assembling laser light scattering instruments from basic building blocks (optical *Lego*®). This allows each of NASA's Principal Investigators (PIs) to tailor the set-up of identical pieces of instrumentation to meet their specific needs, such as dynamic light scattering, static light scattering, or dynamic depolarized light scattering. We believe that modular equipment will encourage a range of applications anywhere from single-angle studies in a simple glovebox apparatus to elaborate, simultaneous multiple-angle studies and beyond.

Crystallization of hard spheres, critical phenomena, nucleation, spinodal decomposition, gelation, aggregation, and diffusion are influenced by gravity and can be studied effectively with LLS in a microgravity environment. LLS can enhance protein crystal growth experiments which need quantitative information about the growth process and an indication of the onset of nucleation. Dynamic light scattering is used for measuring and correlating fluctuations in the intensity of the light

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scattered from particles in Brownian motion. This gives a diffusion coefficient from which particle sizes in a range from 3 nanometers to above 3 microns are derived. Static light scattering is the measurement of the time-averaged intensity scattered by dispersions of particles and macromolecules such as polymers, proteins, micelles, and microemulsions. It is an important tool for determining particle structure, weight-average molecular weight and particle interactions. Dynamic, depolarized laser light scattering examines the weak, horizontally-polarized light scattered from a sample illuminated by a vertically polarized laser beam and derives dynamical and structural information, such as rotational properties and particle aspect ratios, which is not otherwise readily available.

The major goals of the Laser Light Scattering Advanced Technology Development program were to:

- Define and ascertain instrument needs of potential PI's, and to raise consciousness among ground-based LLS users of the unique offerings of the microgravity environment.
- Modify a commonly-available instrument to make it compact, rugged, power efficient, and microgravity-ready.
- Make the LLS instrument modular to allow it to be easily reconfigured and optimized for a wide range of experiments proposed to the NASA Research Announcements (NRA's).
- Automate and enhance data-taking and analysis for the modular LLS instrument.
- Collect data sets on the traditional state-of-the-art, room-size instrument in the NASA Lewis LLS laboratory for evaluation of the hardware and software under development, and to further develop in-house expertise in preparing and analyzing different classes of samples.
- Test miniature LLS modules attached to fiber optic probes with protein crystal growth experiments in collaboration with Professor W.W. Wilson of Mississippi State University/MSFC and demonstrate the LLS instrumentation. Tests were run on microemulsions with Cheung/Miller (U. of Akron), on fractals with Cawley (Case Western Reserve University), and on zeolites with Sacco (Worcester Polytechnic Inst.) and on zeolites with Dutta (Ohio State University) and on protein crystals with Professor Franz Rosenberger/University of Alabama-Huntsville
- Through simulation or actual experiment, test the effects of vibration and low-gravity on system performance.
- Identify enabling technologies and foster their growth. The single card correlator (Brookhaven Instruments Corp.), super-sensitive, miniature detectors and miniature, highly coherent laser diodes (EG&G Canada), and backscatter fiber optic probes (H.S. Dhadwal, SUNY - Stony Brook) have all been sponsored by the LLS ATD project.
- Acquire and evaluate a simultaneous multi-angle LLS instrument.
- Provide assistance to PI's and to NASA's LLS flight hardware project as needed.

Significant Progress

NASA Lewis held an Advanced Technology Development (ATD) Laser Light Scattering Workshop in September of 1988. Major LLS contributors, manufacturers, and potential PI's from around the world attended the workshop. Many issues were settled in formal and informal discussions during the two day workshop. A draft version of the workshop proceedings was edited and issued soon after the workshop and a final, 308-page version of the workshop proceedings (NASA CP-10033) was published August 1989. In January of 1990, more than a dozen potential PIs met to discuss and share their ideas and proposal outlines. At the same time, Dr. Robert G.W. Brown from the Royal Signals and Radar Establishment (RSRE) showed their developments in miniature LLS systems and offered to transfer a large block of RSRE technological advances to NASA.

The project team exhibited the backscatter fiber optic probes (discussed below) at the Microgravity Fluids workshop in August 1990. This provided the opportunity to discuss the LLS project with additional potential PIs and to excite the LLS community with a live demonstration of cutting-edge technology.

Dr. Robert G.W. Brown has served as a hardware consultant on this project. He invented and patented many of the advances needed for making the miniature modules and improving the detectors used in LLS. He has provided detailed lists of the necessary miniature module specifications and procedures for ensuring that these specifications have been met. The core of these specifications were provided in a previous Statement of Work (SOW) to NASA Headquarters. At the January 1990 Washington, D.C. meeting, Dr. Brown, at the time representing the Royal Signals and Radar Establishment, showed RSRE's developments in miniature LLS systems in a presentation to potential PIs. Some of these systems had been tailored for NASA needs, and they were a generation ahead of the rest of the world. The meeting gave potential PIs a chance to look ahead at what was technologically possible, and it gave NASA and RSRE important feedback on how well the needs of the potential PIs were being met. When the commercial portion of RSRE was closed, NASA asked EG&G (then RCA) to license these developments. EG&G has since developed this new generation of detectors for NASA and officially announced them at the August 1992 Photon Correlation and Scattering conference in Boulder, Colorado. This conference was co-sponsored by NASA Headquarters and had several microgravity-related presentations.

Significant advances in backscatter fiber optic probes sponsored by a grant initiated by the LLS ATD project with Professor Harbans Dhadwal at SUNY-Stony Brook have resulted in a patent award for the devices. These fiber optic probes allow LLS to be used in solutions which are opaque to laser light, and hence beyond the interrogation abilities of conventional LLS. We have used the backscatter probes to study milky concentrations ranging up to 10% weight concentration (39 nm particles) without encountering any multiple scattering problems. The probes have proven to be important when LLS is applied to protein crystal growth experiments, as documented in a recent paper by H. S. Dhadwal, W.W. Wilson, R.R. Ansari, and W.V. Meyer entitled "Dynamic light Scattering Studies of BSA and Lysozyme using a Backscatter Fiber Optic System", presented at the Biomedical Optics '93 Conference in Los Angeles (Jan. 1993). The paper discusses detecting the size of BSA and Lysozyme proteins under conditions of varying concentration. It reports LLS measurements which we made on high concentration protein solutions, measurements which have never before been possible because of multiple scattering effects. Our success was due to the backscatter fiber optic probes combined with the new, highly sensitive LLS instrument which has matured under the wings of this ATD project.

In July, we visited Professor Franz Rosenberger at his laboratory at the University of Alabama in Huntsville and worked with him using our briefcase-size LLS laboratory. (Besides the savings in cost and space, this miniature LLS instrument is technically superior to the traditional nine by twelve foot room filled with equipment.) Our visit culminated in a successful demonstration of the new generation correlator and laser/detector module, a fiber optics beam delivery and receiving system using a ninety degree scattering cell, and a pair of GRIN (graded index of refraction) lenses.

Additionally, we have characterized microemulsions with improved results using the noninvasive backscatter probe. R.R. Ansari, H.S. Dhadwal, H.M. Cheung, and W.V. Meyer presented this work in a paper at an international conference on Photon Correlation and Scattering, sponsored by the Optical Society of America and NASA (Boulder, 1992). The paper, entitled "Microemulsion Characterization Using a Fiber Optic Probe" was published in Applied Optics (7/20/93). At this same workshop, Meyer led a discussion session and was then asked to co-organize the next international Photon Correlation meeting to take place in Europe in about two years. At this time, we are planning to make this part of a

NATO / NASA meeting which will introduce the new advances in the microgravity laser light and surface light scattering program along with other advances in photon correlation. We hope this conference will include a hands-on training session and will result in a book of lectures for researchers new to the field. Any NASA HQ involvement/sponsorship has not been determined at this time.

R.R. Ansari and H.S. Dhadwal have authored a set of papers which apply the high-concentration backscatter probe to the detection and characterization of cataracts, work which was made possible through the new technology generated by the LLS ATD project. For example, they presented "A Fiber Optic Probe for the Detection of Cataracts" at TECHNOLOGY 2002 (Dec. 1992). This and similar work by the authors led to an invitation by the National Institute of Health (NIH) to give a presentation in Washington, DC in February, 1993. This presentation was preparatory to setting up clinical trials to use the backscatter probe for the early detection of cataracts in the human eye lens. When the protein aggregates in the eye lens get large enough, they are referred to as a cataract. The ability to monitor this process before and during aggregation will enhance our understanding of cataract growth, and may lead to methods of prevention and/or early treatment.

The work for the single angle LLS instrument is being incorporated into two glovebox experiments flyable aboard the Space Shuttle. The original ATD instrument work is being extended to meet the needs of Professors Paul Chaikin and Wm Russel at Princeton. This compact instrument is capable of measuring properties of a system of hard spheres as they go through a phase transition due to changing concentration. The glovebox LLS instrument will be able to measure the Brownian motion of the fluid, the Bragg scattering of the crystal state, the structure of the glassy (non-ordered) state, and the shear modulus of crystals with dynamic light scattering. An engineering model of this glovebox experiment (CDOT) and one for Dr. Ansari's flocculation experiment (FAME) have been built. Ansari's LLS glovebox hardware is planned for use aboard the Lear Jet for instrument vibration testing. These glovebox units use the new technology advances of the laser light scattering ATD program.

The specifications and verification procedures for the simultaneous multi-angle instrument were sent out in October 1992. This 30+ page document of technical details and equations will be evaluated as a Request for Proposal (RFP) in 1993. We are indebted to Professor David Cannell at UCSB, a consultant for the LLS ATD project for several years, for his contribution to this work. The evaluation teams consist of four groups: the laser light scattering ATD design team, the microgravity flight hardware team, and the two Principal Investigator (PI) teams. This breadboard of optics and simultaneous detectors and lasers will be ideally suited for many of the proposals which have been and will continue to be submitted to the NASA Research Announcements (NRAs) in Fluids, Materials, Fundamental Science, and Biotechnology

The power of the new, multi-angle instrument will be enhanced by the addition of multi-angle algorithms. With these algorithms, a given data set will yield more information, and we anticipate that they will decrease the stiffness of the numerical problem and make it easier to fit histograms to the data sets. The development of the multi-angle code by Professor Cheung of the University of Akron is complete. The code is now consistently extracting bimodal particle size distributions from the data libraries. Professor Cheung's work on these algorithms built upon Cummins and Staples work and was completed in the Summer of 1992 with the aid of NASA's Summer Faculty Fellow program.

The core of correlator control program for single detector input is finished and is being used at Lewis to automatically sample, retrieve and analyze data, and print out the results. A conversion of the program to run under Microsoft Windows needs to be undertaken. This may be handled by the manufacturer (Brookhaven Instruments Corporation) at no cost.

Continuing LLS experiments conducted in the NASA Lewis LLS laboratory have provided many data sets for analysis and testing of both the software and hardware being developed for this ATD effort. While this library of data is needed for evaluating the modular miniaturized hardware in its different configurations, it also perfects in-house expertise in challenging LLS experiment areas. Additional data using backscattering fiber optic probes with protein crystals, microemulsions, etc. were published (see publication list attached). One spin-off of this work is that this instrument will likely be able to non-invasively monitor the sizes of proteins (i.e., cataract development) in human lenses (see "Publications"). We have attempted to characterize zeolites with Professor A. Sacco of Worcester Polytechnic Inst. The work with Sacco is incomplete at this time, because it will be necessary to polymerize the end of the probe to use it in the caustic material Sacco uses in his experiments. The use of the backscatter probe has also allowed us to study the transitions from single to multiple scattering. This may prove important for PIs requesting diffusing wave spectroscopy measurements (i.e., multiple scattering).

Assistance has been provided to the STDCE (Surface Tension Driven Convection Experiment) flight hardware project when it is in need of LLS diagnostics. This was discussed in a previous report.

Additionally, NASA technical knowledge has been transferred to the ESA Critical Point Facility team at the request of MSAD (Roger Crouch) and NASA LeRC Space Experiments Division (Al Wilkinson). This occurred in April 1991.

The attached milestone chart gives a concise history of this Laser Light Scattering Advanced Technology Development Project. The complete final report submitted to NASA Headquarters contains, in addition to the list of publications that have resulted from this work, a complete set of publications attached as an appendix.

LASER LIGHT SCATTERING ATD

Publications and Presentations

1. "Proceedings NASA Laser Light Scattering Advanced Technology Development Workshop, 1988", CP10033, published August 1989, edited by W.V. Meyer.

2. Dhadwal, H.S., "Regualrized analysis of correlation functions - The particle size distribution, Proceedings of the European Symposium on Particle Characterization", Nuremberg, April 19-21, 1989. In support of NASA LeRC grant NAG 3-963.

3. Dhadwal, H.S., Wu, C., and Chu, B., "Fiber optic detector probes for laser light scattering", Appl Opt 19, 4199-4205 (1989). In support of NASA LeRC grant NAG 3-963.

4. Ross, D.A. and Dhadwal, H.S., "Regularized inversion of the Laplace transform: accuracy assessed theoretically and by simulation", Proceeding of the 2nd International Congress on Optical Particle Sizing, Arizona, March 5-8, (1990). In support of NASA LeRC grant NAG 3-963.

5. Dhadwal H.S., Ansari, R.R., and Meyer, W.V., "Demonstration of a fiber optic probe for sizing colloidal dispersions", NASA Fluid Physics Microgravity Workshop, Cleveland, OH., August 1990.

6. Dhadwal, H.S. and Ross, D.A., "Particle characterization using successive low pass filtering", Proceedings of Second World Congress Particle Technology, September 19-22, 1990, Kyoto, Japan. In support of NASA LeRC grant NAG 3-963.

7. Meyer, W.V. and Ansari, R.R., "A Preview of a Microgravity Laser Light Scattering Instrument", AIAA 91-0779. This paper was presented January 9, 1991 at the 29th Aerospace Sciences Meeting.

8. Dhadwal, H.S., Ansari, R.R., and Meyer, W.V., "A Fiber Optic Probe for Particle Sizing in Concentrated Suspensions", Review of Scientific Instruments **62** (12), December 1991. In support of NASA LeRC grant NAG 3-172.

9. Ross, D.A. and Dhadwal, H.S., "Regularized Inversion of the Laplace Transform: Accuracy of Analytical and Discrete Inversion", Particle Particle system Characterization, 8, 282-286 (1991). In support of NASA LeRC grant NAG 3-963.

10. Dhadwal, H.S., "A back Scatter Fiber Optic Probe For Particle Sizing", Gradient-Index Optical Systems Topical Meeting, Topical Meeting, Monterey, CA. April 8-9, (1991). In support of NASA LeRC grant NAG 3-172.

11. Ansari, R.R., Meyer, W.V., and Dhadwal, H.S., "Particle Sizing of Concentrated Suspensions Using a Back Scattered Fiber Optic Probe", Paper No. 92, presented at 65th Colloid and Surface Science Symposium, June 17-19, 1991, Norman, Oklahoma.

12. Ansari, R.R., Ali-Kahn, A., Meyer, W.V., "Static and Dynamic Laser Light Scattering Studies of Nylon 6,6 in Trifluoroethanol Containing Sodium Trifluoracetate", Paper No. 196, presented at 65th Colloid and Surface Science Symposium, June 17-19, 1991, Norman, Oklahoma.

13. Dhadwal, H.S., Ansari, R.R., "Multiple Fiber Optic Probe For Several Sensing Applications", in Fiber Optic and Laser Sensors IX , Volume **1584-34**, SPIE's OE/FIBERS '91 Symposium, Boston, MA, September 3-6, 1991.

14. Gay, T.J., Khakoo, M.A., Brand, J.A., Furst, J.E., Meyer. W.V., Wijayarathna, W.M.K.P., Dunning, F.B., "Extrapolation Procedures in Mott Electron Polarimetry", Review of Scientific Instruments 63(1), January 1992. Supported by NSF.

15. Ansari, R.R., Dhadwal, H.S., Campbell, M.C.W., and Dellavecchia, M.A., "Fiber Optic Sensor for Ophthalmic Refractive Diagnostics", in Fiber Optic Medical and Fluorescent Sensors and Applications, Volume **1648**, pages 83-105, Progress in Biomedical Optics, Editor Abraham Katzir, SPIE Publication 1992.

16. Ansari, R.R., Dhadwal, H.S., Cheung, H.M., and Meyer, W.V., "A Fiber Optic Probe for Characterization of Microemulsions", 9th International Symposium on Surfactants in Solution, June 10-15, 1992, Varna, Bulgaria.

17. Ansari, R.R., Dhadwal, H.S., Cheung, H. M., and Meyer, W.V., "Use of a Fiber Optic Probe for Quasi-Elastic Light Scattering Measurements in Microemulsions", 66th Colloid & Surface Science Symposium, paper # 41, June 14-17, 1992, Morgantown, West Virginia. In support of NASA LeRC grant NCC3-240.

18. Rim, Y.H., Cawley, J.D., Ansari, R.R., Meyer, W.V., "In-Situ Light Scattering Study of Aggregation", in Ceramic Transactions, Forming Science and Technology for Ceramics, Vol. 26, pp. 54-65, edited by J.J. Cima, 1992 as part of the Proceedings for 93rd Annual American Ceramic Society Meeting.

19. Ansari, R.R., Dhadwal, H.S., Cheung, H. M., and Meyer, W.V., "Microemulsion Characterization Using a Fiber Optic Probe", J. App. Optics, Vol. 32, No. 21, July 20, 1993, presented at Photon Correlation and Scattering (Theory and Applications) 8th Topical Meeting, Optical Society of America, August 24-26, 1992, Boulder, Colorado. In support of NASA LeRC grants NAG 3-172 and NCC3-240.

20. Dhadwal, H.S., Khan, R.R., Suh, K., "An Integrated Fiber Optic Probe for Dynamic Light Scattering", J. App. Optics, Vol. 32, No. 21, July 20, 1993, Photon Correlation and Scattering (Theory and Applications), Poster # PD2, 8th Topical Meeting, Optical Society of America, August 24-26, 1992, Boulder, Colorado. In support of NASA LeRC grant NCC3-241.

21. Dhadwal, H.S., Wilson, W.W., Ansari, R.R., and Meyer, W.V., "Dynamic Light Scattering Studies of BSA and Lysozyme using a Backscatter Fiber Optic System", Proceedings of Static and Dynamic Light Scattering in Medicine and Biology, Biomedical Optics '93, 16-23, January 21-22, 1993, Los Angeles, CA. In support of NASA LeRC grant NAG 3-172.

22. Ansari, R.R. and Dhadwal, H.S., "A Fiber Optic Probe for the Detection of Cataracts", TECHNOLOGY 2002: The Third Technology Transfer Conference and Exposition, December 1-3, 1992, Baltimore, MD, NASA CP 3189, Feb. '93.

23. Dhadwal, H.S. and Ansari, R.R., "A Coherent Fiber Optic Sensor for Early Detection of Cataractogenesis in a Human Eye Lens", submitted to Optical Engineering: Biomedical Optics Special Issue - February 1993. In support of NASA LeRC grant NCC 3-241.

Technical Reports

1. Ansari, R.R., and Meyer, W.V., "Laser Light Scattering Studies on the Interactions and Characterization of Alumina and Pliolite Plastic Particles in Silicone Oil", Space Experiments Division Technical Report, NASA-LeRC, April 1991.

2. Ansari, R.R., and Meyer, W.V., "Static and Dynamic Laser Light Scattering Studies of Series 78 Nylon 6,6 Polymer", NASA/Du Pont Confidential Divisional Report, April 1991.

Patents

1. United States Patent Number 5,028,135, H.M. Cheung, issued Jul. 2, 1991, "Combined High Spatial Resolution and High Total Intensity Selection Optical Train for Laser Spectroscopy", (continuation in part of U.S. Patent Appl. 07/403,096)

2. United States Patent Number 5,155,549, Harbans S. Dhadwal, issued October 13, 1992, "Method and Apparatus for Determining the Physical Properties of Materials Using Dynamic Light Scattering Techniques."

3. Method and Apparatus for Determining the Physical Characteristics of Ocular Tissue. (Inventors: R.R. Ansari and H.S. Dhadwal), Applied for January 23, 1992, issued July 15, 1993, docket #479-2. We are still waiting for notification of the patent number.

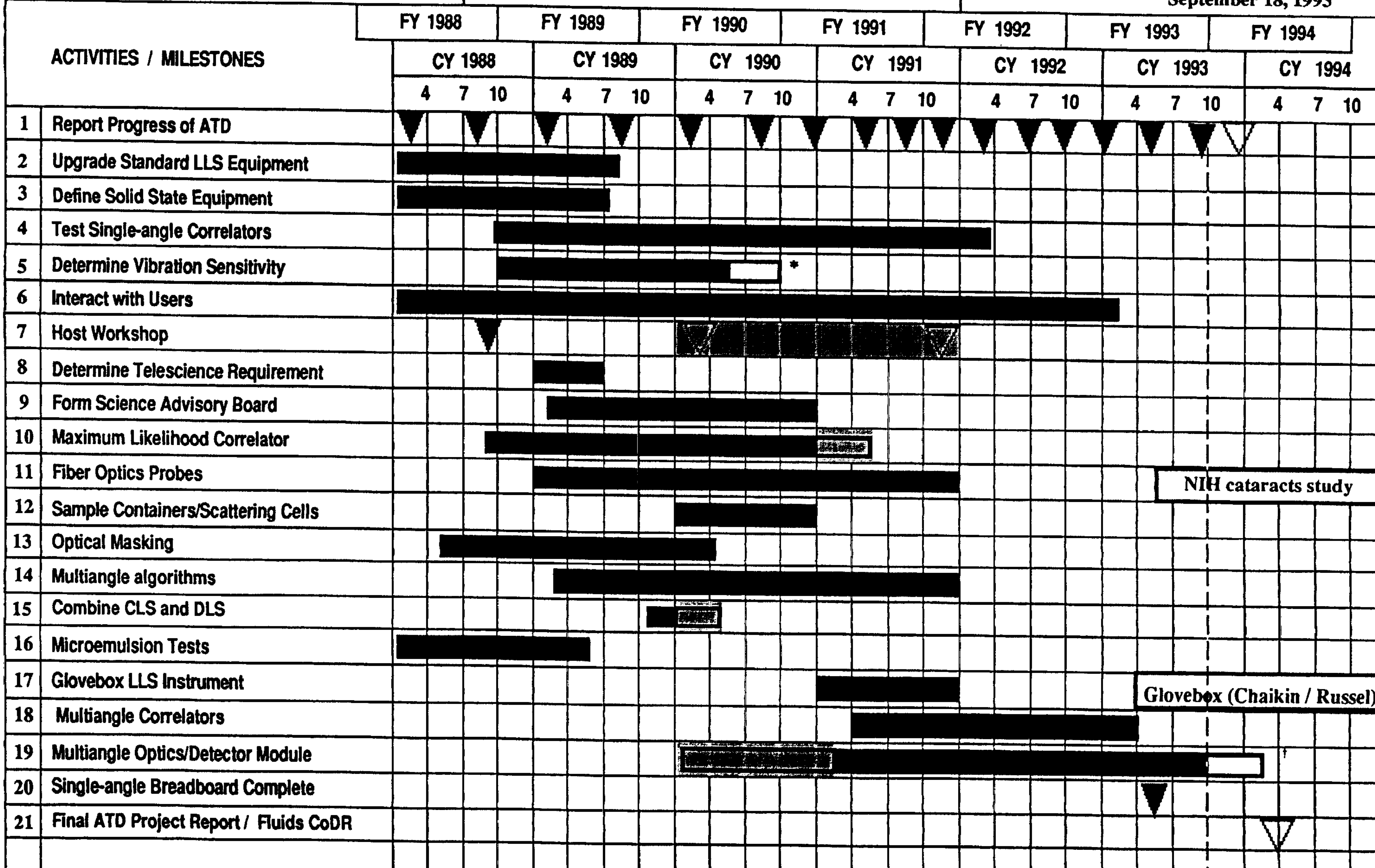
APPROVAL RESPONSIBILITY

ACCOMPLISHMENT RESPONSIBILITY
William V. Meyer

MASTER PROGRAM / PROJECT SCHEDULE

LEWIS RESEARCH CENTER
LASER LIGHT SCATTERING INSTRUMENT

ORIGINAL SCHEDULE APPROVAL
LAST SCHEDULE CHANGE March 27, 1993
STATUS AS OF September 18, 1993



* Scheduled for Lear Jet test of Ansari's FAME glovebox

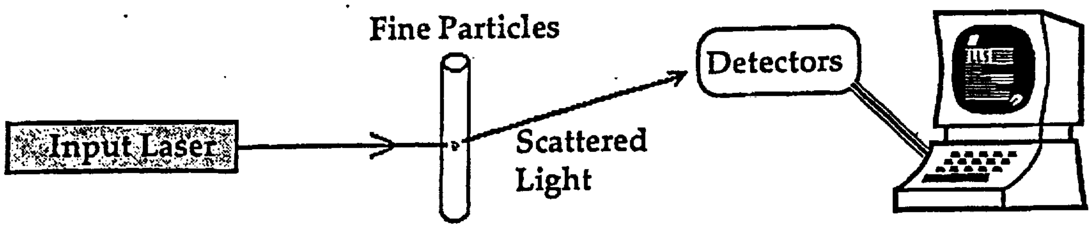
■ = Activities curtailed by budget change

† Transferred to flight proj.

MICROGRAVITY MATERIALS SCIENCE LABORATORY

NASA Lewis Research Center

Laser Light Scattering Advanced Technology



This instrument will enable the following kinds of research:

(1) Hard-sphere interactions,
(2) Aggregation,
(3) Diffusion,
(4) Critical phenomena,
(5) Nucleation,
(6) Microemulsions,
(7) Spinodal decomposition,
(8) and others.

LeRC team leads a worldwide development program to prepare a versatile flight instrument.

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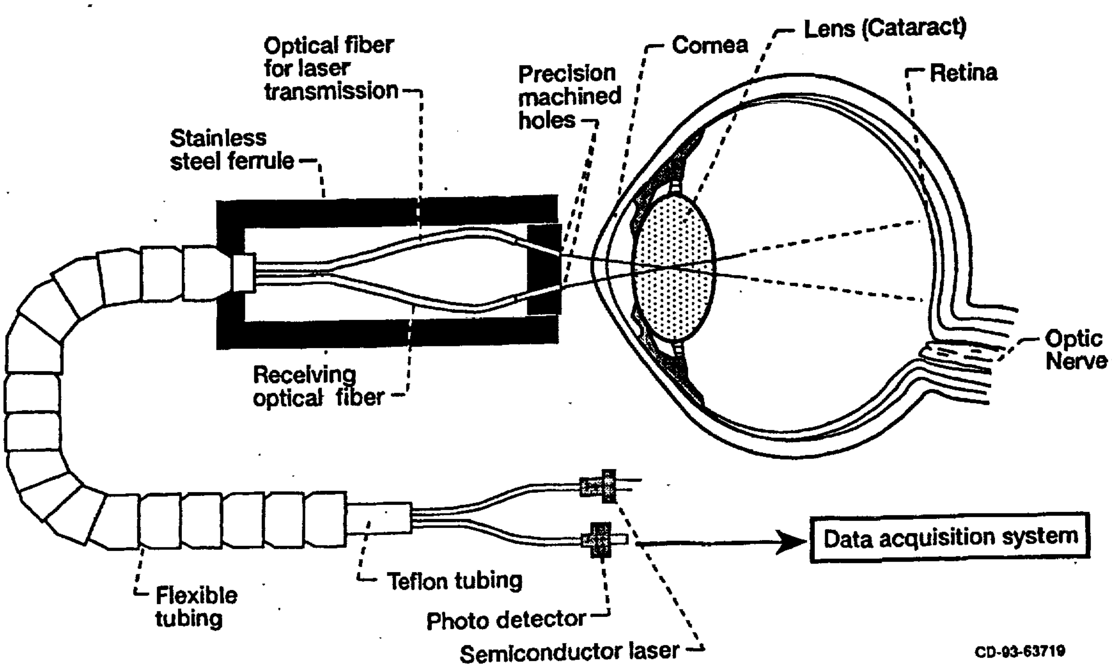
A FIBER OPTIC SYSTEM FOR EARLY DETECTION OF CATARACTS



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