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INFRARED PROPERTIES OF HIGHLY TRANSPARENT SOLIDS.(U)
JUL 80 M BRAUNSTEIN F49620-78-C-0094

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The optical properties of highly transparent single crystals of KBr, KCl, CaF_2 and BaF_2 have been measured using laser calorimetric techniques and wavelength modulation spectroscopy techniques in the spectral range from 2.5 to 12 microns in cooperation with the University of California at Los Angeles (UCLA). Rich and varied absorption structures in

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the spectra were observed in all of the crystals enabling an identification of volume and surface absorption. Similar dominant bands are observed in many of the crystals indicating the presence of common impurities regardless of the crystal and the origin of its growth. However, varied fine structure was observed in different crystals which are indicative of the individual characteristics of the chemistry of the crystal preparation. The measurements were performed in laboratory and dry nitrogen ambients and the physical absorption and desorption of surface contaminants was readily observed.

Ellipsometric techniques were developed to evaluate and measure the strain and anisotropy in infrared optical films. The data processing and analysis techniques which were developed allowed, for the first time, the evaluation of multilayer thin film systems for strain, anisotropy and surface contaminant layers. The importance of good quality and properly prepared film samples was shown to be of prime importance in order to obtain consistent results in the data ananlysis.

Improved Reactive Atmosphere Processing, (RAP), techniques were developed and studied using a two zone RAP furnace for single crystal growth of BaF_2 and CaF_2 which resulted in improved infrared transmission in the 3 to 5 micron wavelength region for the first time.

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INFRARED PROPERTIES
OF
HIGHLY TRANSPARENT SOLIDS

Final Report

Period Covered: July 1, 1978 to July 1, 1980

Contract No: F49620-78-C-0094

Prepared for:

Air Force Office of Scientific Research
Bolling Air Force Base, D.C. 20332

Prepared By:

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RESEARCH OBJECTIVES:

The objectives of this research program were:

a. Study the residual absorption in high purity materials which are highly transparent in the infrared spectral region;

b. Investigate the optical, mechanical and structural properties of infrared transparent thin films and coatings;

c. Study methods to passivate and harden the surfaces of bulk materials grown by the Reactive Atmosphere Process (RAP);

d. Calculate fundamental extrinsic loss mechanisms in bulk materials and thin films subjected to intense infrared lasers.

STATUS OF THE RESEARCH EFFORT:

This report summarizes the significant accomplishments of the research on the study of the Infrared Properties of Highly Transparent Solids.

Infrared Wavelength Modulation Spectroscopy

In cooperation with the group at the University of California at Los Angeles, we performed infrared laser calorimetric measurements on single crystal samples of CaF_2, LiF, NaCl, NaF, LaF_3, BaF_2, MgF_2, SrF_2 and MgO. The measurements provided calibration points to enable wavelength modulation spectroscopy to be performed on the samples in the spectral region from 2.5 to 12 μm. This has enabled, for the first time, the measurement of the optical absorption at levels of 10^{-5} cm^{-1} on highly transparent solids. Previously, only measurements at discrete wavelengths, by thermocouple laser calorimetry and photoacoustic calorimetry, were available which limited the ability to adequately measure (in a simple manner) the surface and volume absorption of infrared transparent materials. The ability to study mechanisms and causes for the limitations to infrared transparency of currently available alkali halide and alkaline earth fluoride materials was also restricted. The aforementioned class of materials has important applications in a variety of Department of Defense and Department of Energy high-energy laser systems which require transparent windows which can withstand high laser fluxes.

The rich and varied absorption spectra observed using wavelength modulation spectroscopy on the crystal samples enabled the identification of volume and surface absorption at levels of 10^{-4} to 10^{-5} cm^{-1}, which indicates that a fraction of a monolayer of surface absorbed species can be detected. Future experiments in controlled ambients on well-characterized substrates using this technique can be rewarding for the study of the physics and chemistry of surfaces and should lead to a better understanding of surface catalysis and semiconductor surface states or interfaces.

The growth, preparation and initial characterization by laser calorimetry of the single crystal samples of high purity and high infrared transparency at Hughes Research Laboratories, for use in the wavelength modulation spectroscopy experiments performed at UCLA, were of critical importance in permitting the demonstration of the ability to measure absorption at levels of 10^{-5} cm^{-1}.

Ellipsometric Measurements

In our study of the strain and anisotropy in infrared optical films using ellipsometry, we have demonstrated, for the first time, that ellipsometry can be used effectively for investigating these properties of films in single layer and multilayer forms. In addition, our development of the computer data processing analysis of the experimental data taken from the ellipsometric measurements on films of As_2S_3 and As_2Se_3 has demonstrated, also for the first time, that we can measure the optical constants accurately for metals, such as aluminum,or semiconductors, such as silicon or gallium arsenide, in an ordinary laboratory ambient, where one knows that surface contaminants due to reaction with oxygen and water vapor prevented previous workers from getting accurate measurements. These results are extremely important since previously one would have thought that it was necessary to do in situ measurements in a clean ultra-high vacuum system for highly reactive sample surfaces such as aluminum or silicon.

Reactive Atmosphere Processing

Reactive Atmosphere Processing (RAP), developed at Hughes Research Laboratories on earlier contract efforts supported by the Defense Advanced Research Projects Agency as an extension of our Internal Research and Development RAP discoveries, has made major contributions to the improvement in the infrared transparency of KCl, CaF_2 and BaF_2. However, we knew that further improvements, particularly for CaF_2,could be achieved with additional study. This is because the higher crystal growth temperature required for CaF_2 (compared to KCl) permits excessive reactivity of the CaF_2 with outgassing hydroxyl ions from the growth-furnace walls. During this program effort, a two-chamber furnace was constructed which permitted us to confine the crystal growth region to a smaller volume than used in our earlier work. This allowed us to inject the RAP agent (HF) directly into the growth zone to more effectively clean up the growing CaF_2 crystal. This procedure was not effective in the previously used single-chamber furnace because of the large surface area of graphite felt,surrounding the graphite heating elements,which can be a source of moisture-associated impurities, e.g., OH^-. In addition, less reaction time for pre-processing the starting material by RAP was required when the two-chamber furnace was used. Our experiments under this program conclusively show that the two-zone RAP furnace removes the residual absorption in the 3 to 5 μm spectral region for CaF_2, which we observed in crystals prepared in the old (single chamber) furnace. This new high-purity CaF_2 should be of great interest and importance to HF-DF high energy laser system developers.

Publications

"Strain Induced Anisotropy in As_2S_3, As_2Se_3, and ZnSe Films on KCl Substrates via 10.6 μm and 0.6328 μm Ellipsometer Measurements and 0.6328 μm Reflector Measurements", M.E. Pedinoff, M. Braunstein and O.M. Stafsudd, Applied Optics, Volume 18, No. 2, January 15, 1979.

"The Effect of Anisotropy on the Measurement of the Refractive Index of Thin Transparent Films", M.E. Pedinoff, M. Braunstein, and O.M. Stafsudd, presented at the Fourth International Conference on Ellipsometry, Lawrence Berkeley Laboratory, Berkeley, California, August 20-23, 1979, to be published in Conference Proceedings.

"Wavelength Modulation Spectroscopy of Highly Transparent Solids", M. Braunstein, R. Braunstein, and R.K. Kim, presented at the Eleventh Annual Symposium on Optical Materials for High Power Lasers, NBS, Boulder, Colorado, October 30-31, 1979, published as special publication by NBS as the Conference Proceedings, NBS Publication 568.

"Scattering Losses in Single and Polycrystalline Infrared Materials for Infrared Fiber Application", J.A. Harrington, M. Braunstein, B. Bobbs and R. Braunstein (to be published in Proceedings of American Ceramic Society, Annual Meeting, April 1980, Volume on Physics of Optical Fibers, 1980).

"Infrared-Transparent Glasses Derived from the Fluorides, Thorium, Barium", M. Robinson, R.C. Pastor, R.R. Turk, D.P. Devor, M. Braunstein and R. Braunstein, Mat. Res. Bull. 15, 735 (1980).

"Infrared Wavelength Modulation Spectroscopy of Laser Window Materials", R. Braunstein, R.K. Kim and M. Braunstein, presented at Boulder Damage Symposium, September 30, 1980, to appear in NBS Proceedings of the Conference.

Professional Personnel Associated with the Research

M. Braunstein	Principal Investigator
J.A. Harrington	Laser Calorimetry
R.C. Pastor	RAP Crystal Growth
M.E. Pedinoff	Ellipsometry
J.E. Rudisill	Optical Films

Scientific Interactions

Collaboration was maintained between the research effort at Hughes Research Laboratories (HRL) and the research effort at the University of California at Los Angeles (UCLA). Laser calorimetric measurements at HRL, in combination with wavelength modulation spectroscopy evaluation measurements (at UCLA), were performed on a variety of samples in the optical region between 2.5 to 12 μm.

The HRL group supplied several single crystal samples of alkali halides and alkaline earth fluorides to the research group at the University of Southern California (USC) during this research effort.

M. Braunstein presented an invited paper entitled, "Thin Film Materials, UV and IR" at the 10th Annual Damage Symposium, September 12-14, 1978, NBS, Boulder, Colorado.

J.A. Harrington presented an invited paper entitled, "State-of-the-Art of IR Window Materials at the above-referenced Damage Symposium.

Patents or New Discoveries

None.

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