

20000920302

REPORT DOCUMENTATION PAGE

AD-A262 361

Form Approved
OMB No. 0704-0188

(2)

It takes an average of 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering the collection of information. Send comments regarding this burden estimate or any other aspect of this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. REPORT DATE

3. REPORT TYPE AND DATES COVERED

Final Report 15 Jun 91 - 14 Jun 92

5. FUNDING NUMBERS

DARPA

DEVELOPMENT OF MODEL-BASED MAGNETIC LP-LEC GROWTH OF
LARGE DIAMETER GAAS

6. AUTHOR(S)

Professor August F. Witt

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Department of Materials Science & Engineering
Massachusetts Institute of Technology
77 Massachusetts Avenue
Cambridge, MA 02139-4307

AFOSR-TR-

8. PERFORMING ORGANIZATION
(REPORT NUMBER) 9

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

AFOSR/NE
110 Duncan Avenue Suite B115
Bolling AFB, DC 20332-0001

Dr. Witt

10. SPONSORING/MONITORING
AGENCY REPORT NUMBER

AFOSR-91-0355

11. SUPPLEMENTARY NOTES

DTIC
ELECTE
APR 01 1993
S E D

12a. DISTRIBUTION / AVAILABILITY STATEMENT

UNLIMITED

12b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

The objective of this research was to explore the range of analytical information on the defect structure of doped and semi-insulating GaAs obtainable from computational, non-invasive, near infrared absorption analysis. Motivation for this research was provided by the realization that the establishment of meaningful property specifications for device materials is contingent on non-invasive defect analysis executable in a fabline environment. Infrared absorption measurements on a micro- and macro-scale in combination with computational image processing and analysis were found to meet the requirements of the stated research objectives.

98 3 31 076

93-06637



14. SUBJECT TERMS

15. NUMBER OF PAGES

16. PRICE CODE

UNCLASSIFIED UNCLASSIFIED UNCLASSIFIED
17. SECURITY CLASSIFICATION OF THIS PAGE
18. SECURITY CLASSIFICATION OF ABSTRACT

20. LIMITATION OF ABSTRACT

UL

GENERAL INSTRUCTIONS FOR COMPLETING SF 298

The Report Documentation Page (RDP) is used in announcing and cataloging reports. It is important that this information be consistent with the rest of the report, particularly the cover and title page. Instructions for filling in each block of the form follow. It is important to *stay within the lines* to meet optical scanning requirements.

Block 1. Agency Use Only (Leave blank).

Block 2. Report Date. Full publication date including day, month, and year, if available (e.g. 1 Jan 88). Must cite at least the year.

Block 3. Type of Report and Dates Covered. State whether report is interim, final, etc. If applicable, enter inclusive report dates (e.g. 10 Jun 87 - 30 Jun 88).

Block 4. Title and Subtitle. A title is taken from the part of the report that provides the most meaningful and complete information. When a report is prepared in more than one volume, repeat the primary title, add volume number, and include subtitle for the specific volume. On classified documents enter the title classification in parentheses.

Block 5. Funding Numbers. To include contract and grant numbers; may include program element number(s), project number(s), task number(s), and work unit number(s). Use the following labels:

C - Contract	PR - Project
G - Grant	TA - Task
PE - Program Element	WU - Work Unit Accession No.

Block 6. Author(s). Name(s) of person(s) responsible for writing the report, performing the research, or credited with the content of the report. If editor or compiler, this should follow the name(s).

Block 7. Performing Organization Name(s) and Address(es). Self-explanatory.

Block 8. Performing Organization Report Number. Enter the unique alphanumeric report number(s) assigned by the organization performing the report.

Block 9. Sponsoring/Monitoring Agency Name(s) and Address(es). Self-explanatory.

Block 10. Sponsoring/Monitoring Agency Report Number. (If known)

Block 11. Supplementary Notes. Enter information not included elsewhere such as: Prepared in cooperation with...; Trans. of...; To be published in.... When a report is revised, include a statement whether the new report supersedes or supplements the older report.

Block 12a. Distribution/Availability Statement. Denotes public availability or limitations. Cite any availability to the public. Enter additional limitations or special markings in all capitals (e.g. NOFORN, REL, ITAR).

DOD - See DoDD 5230.24, "Distribution Statements on Technical Documents."

DOE - See authorities.

NASA - See Handbook NHB 2200.2.

NTIS - Leave blank.

Block 12b. Distribution Code.

DOD - Leave blank.

DOE - Enter DOE distribution categories from the Standard Distribution for Unclassified Scientific and Technical Reports.

NASA - Leave blank.

NTIS - Leave blank.

Block 13. Abstract. Include a brief (Maximum 200 words) factual summary of the most significant information contained in the report.

Block 14. Subject Terms. Keywords or phrases identifying major subjects in the report.

Block 15. Number of Pages. Enter the total number of pages.

Block 16. Price Code. Enter appropriate price code (NTIS only).

Blocks 17. - 19. Security Classifications. Self-explanatory. Enter U.S. Security Classification in accordance with U.S. Security Regulations (i.e., UNCLASSIFIED). If form contains classified information, stamp classification on the top and bottom of the page.

Block 20. Limitation of Abstract. This block must be completed to assign a limitation to the abstract. Enter either UL (unlimited) or SAR (same as report). An entry in this block is necessary if the abstract is to be limited. If blank, the abstract is assumed to be unlimited.

Buck

118

DEVELOPMENT OF MODEL BASED MAGNETIC LP-LEC GROWTH
OF LARGE DIAMETER GaAs

Final Technical Report

AFOSR-91-0355

Period: 06/15/91 - 06/14/92
Amount: \$248,870

Sponsored by:

Defense Advanced Research Projects Agency
Dr. Jane Alexander
703-696-2233

Monitored by AFOSR under Contract No. AFOSR-91-0355

Submitted by:

August F. Witt
Department of Materials Science & Engineering
Massachusetts Institute of Technology
77 Massachusetts Avenue
Cambridge, MA 02139-4307
617/253-5303

Accession For	
NTIS	CRA&I ☒
DTIC	TAB ☐
Unannounced ☐	
Justification	
By	
Distribution /	
Availability Codes	
Dist	Avail and/or Special
A-1	

The views and conclusions contained in this document are those of the authors and should not be interpreted as necessarily representing the official policies or endorsements, either expressed or implied, of the Defense Advanced Research Projects Agency or the U.S. Government.

ACKNOWLEDGMENTS

The principal investigators and support staff associated with this research effort express their gratitude to Dr. J. Alexander for her support. Acknowledged also is the continuing encouragement and help provided by Dr. G. Witt (AFOSR) who monitored with insight and care.

CONTENTS

Acknowledgments.....	i
Research Staff Associated with this Research Effort.....	1
INTRODUCTION.....	1
SUMMARY.....	2
APPROACH TO RESEARCH.....	3
RESULTS.....	7
CONCLUSIONS.....	15
Publications.....	16
Presentations.....	16

STAFF ASSOCIATED WITH THIS RESEARCH EFFORT

Principal Investigator	A.F. Witt
Research Associate	M.J. Wargo
Research Staff	C. Counterman
Graduate Student	X.Z. Cao
Technician	J. DiFrancesco
Secretary	G. Landahl

INTRODUCTION

The objective of this research was to explore the range of analytical information on the defect structure of doped and semi-insulating GaAs obtainable from computational, non-invasive, near infrared absorption analysis. Motivation for this research was provided by the realization that the establishment of meaningful property specifications for device material is contingent on non-invasive defect analysis executable in a fabline environment. Infrared absorption measurements on a micro- and macro-scale in combination with computational image processing and analysis were found to meet the requirements of the stated research objectives.

SUMMARY

Optical semiconductor characterization by computational NIR absorption analysis has been found to be an effective approach to the determination of the following properties:

- dopant concentration and distribution (macroscale)
- local segregation effects (microscale)
- residual macro-stress
- dislocation density and distribution
- stress, associated with dislocations
- precipitates
- annealing effects
- subsurface damage

The broad spectrum of defect identification was achieved by complementing conventional bright field transmission microscopy with dark field, phase contrast and polarized light microscopy.

The approach taken is characterized by its non-invasive nature, applicability on a macro- and micro-scale, high resolution, rapid execution and digital data storage with spatial coordinates suitable to conduct subsequent correlation analyses with spatial distribution of device deficiencies.

APPROACH TO RESEARCH

The analytical technique developed for rapid, non-destructive defect analysis in semiconductors is based on quantitative image analysis in conjunction with near infrared (NIR) transmission microscopy. NIR microscopy provides an image of the semiconductor sample (0.5-2 mm thick) reflecting any local variations in the absorption coefficient across the field due to the presence of defects. Qualitatively this image provides an excellent representation of material uniformity. It contains in addition, however, information which can be used for the quantitative determination of local free charge carrier concentrations and lattice stress conditions, for example.

NIR microscopy relies on an imaging device such as a CCD camera or a silicon vidicon camera to detect transmitted radiation. To quantify the image, the output of the camera is used as input to a digital image processor where the signal is digitized into a 512 by 480 pixel array with a dynamic range of 256 gray levels. The image processing system provides for multiple image storage and near real time whole image mathematics capability. The approach taken for data calibration is to increase the gain of the system so that the entire range is distributed over the limited transmittance range exhibited by the sample; using neutral density filters, the measured gray level value can then be equated to transmittance. Comprehensive defect mapping can be accomplished by determination of the IR (and, as applicable, the visible) spectrum for a given material by means of an FTIR spectrometer and identification of chemical defects, for example, through their characteristic absorption peaks (fig. 1). The spatial distribution of these defects is subsequently analyzed and the corresponding data are stored in digitized form with their spatial coordinates. The fundamental characteristics of computational optical defect analysis are presented in fig. 2. The mode is non-invasive, applicable to both the micro- and macro-scale with a maximum spatial resolution approaching 1 μm ; data storage with

spatial coordinates is provided for with complete image analysis requiring a fraction of one second.

Computational image analysis has been applied to dopant concentration analysis, stress analysis, dislocation density measurements in conducting and semi-insulating matrices, surface damage measurements and precipitation analyses. Work is in progress on the determination of lattice damage associated with ion implantation and on the identification of defect propagation into epitaxial layers.

The non-destructive nature of the computational image analysis in combination with digital data storage provides a means for the conduct of a statistical correlation analysis between device and spatially coincident wafer characteristics. Such analyses can be accomplished upon subjecting the analyzed wafer to device processing and mapping the intermediate processing steps as well as the spatial distribution of yield and performance of devices (fig. 3).

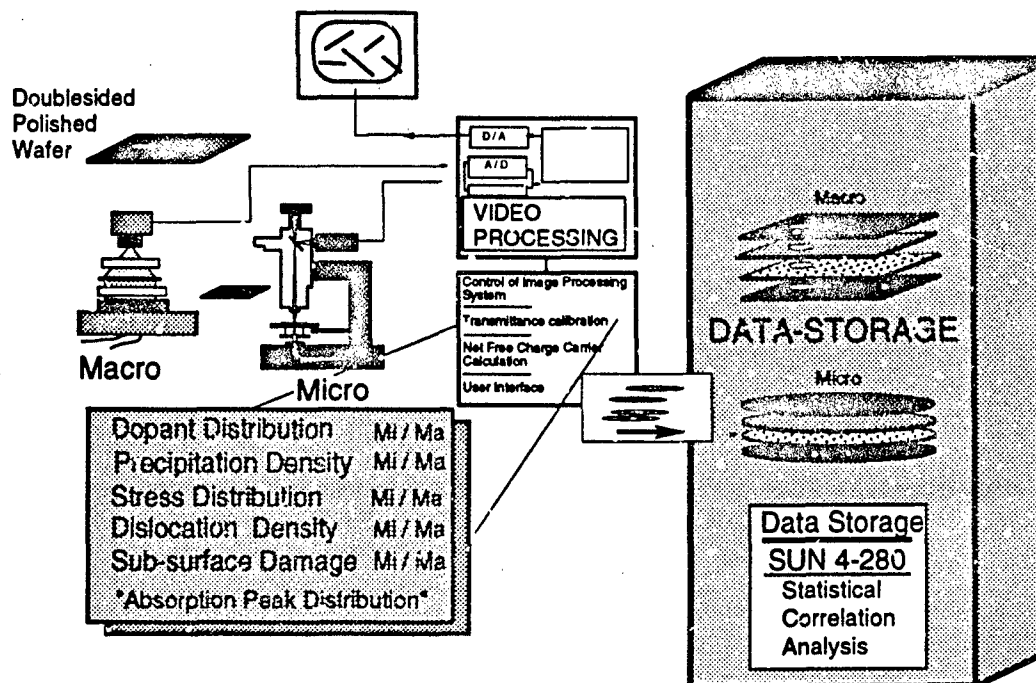
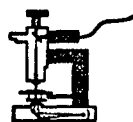


Fig.1 Computational NIR Absorption Analysis with Data Storage

Characteristics:

- * Mode: Non-Invasive
- * Scale: Macro, Selected Micro
- * Resolution: (as applicable) Micron to Sub-Micron Range
- * Data Storage: Digital with Spatial Coordinates
- * Analysis Time: Fractional Seconds

Properties revealed by non-invasive Optical analysis:



Dopant Concentration and Distribution (macro)
 Local Segregation Inhomogeneities (micro)
 Residual Stress Distribution (macro)
 Dislocations (micro)
 Stress Associated with Dislocations (micro)
 Precipitates (micro)
 Surface Damage (macro, micro)
 Annealing Effects (macro, micro)
Absorption peaks in visible and NIR range

Fig.2 Capabilities of Semiconductor Characterization by Computational Absorption Analysis

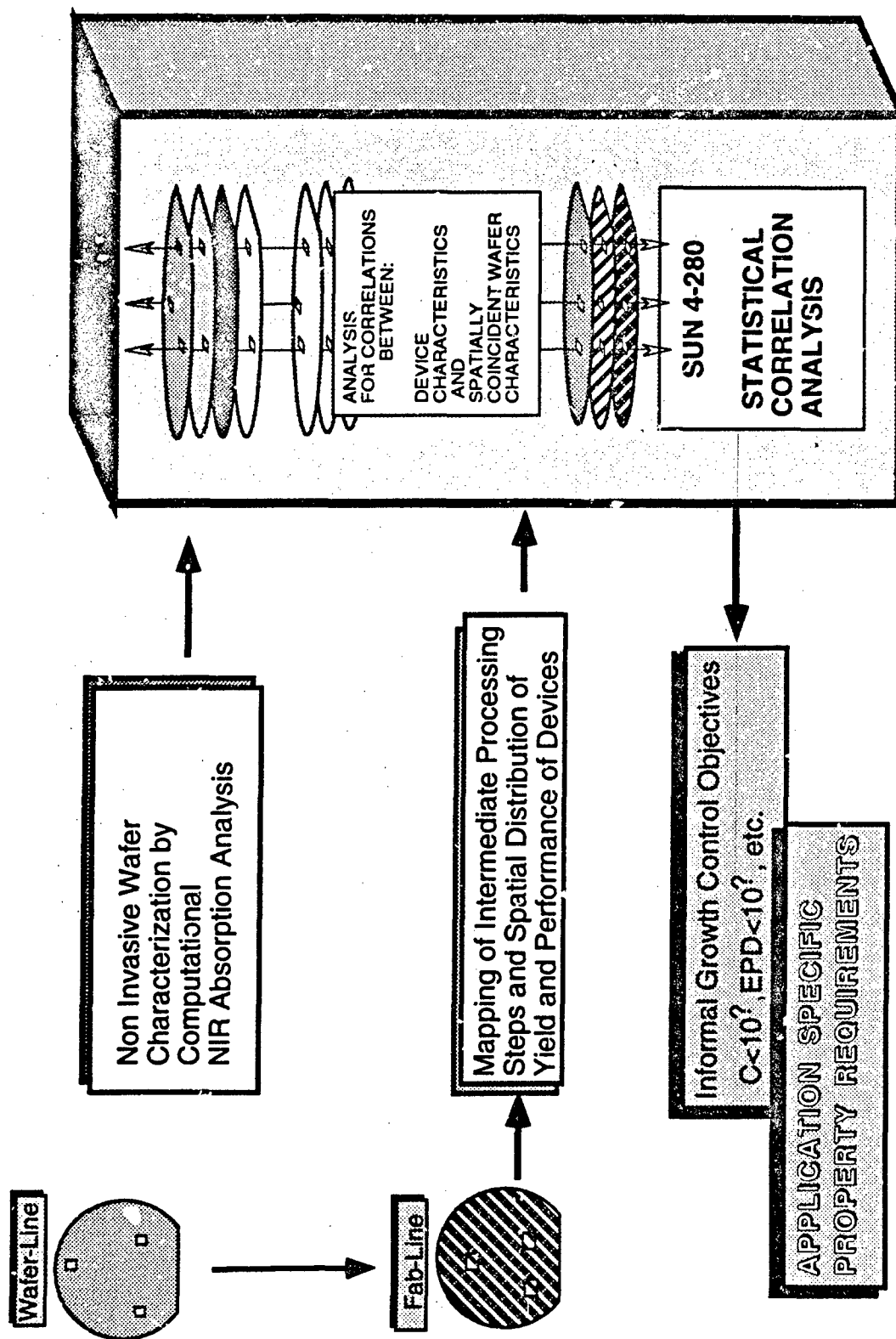


Fig. 3 Establishment Of User Specified Property Requirements Through Statistical Correlation Analysis

RESULTS

An NIR transmission micrograph of commercial Te-doped GaAs with a corresponding free charge carrier (dopant) distribution analysis is given in fig. 4. (The section analyzed was cut from a crystal along its growth axis.) The quantitative microscale analysis reveals that microsegregation effects associated with LP-LEC growth of GaAs result in compositional fluctuations which, over micron dimensions, approach and in some instances exceed one order of magnitude. This finding is contrasted by the general belief that such fluctuations are in the range of ± 15 to 30% of the average doping value. It is also found that, contrary to expectation, the striation pattern is discontinuous along the crystal diameter. On the basis of this quantitative segregation analysis, it is concluded that growth is subjected to turbulent free density driven melt convection; this results in localized thermal perturbations which penetrate the solute boundary layer and lead to melt-back. Subsequent regrowth and dopant incorporation appear largely controlled by fluid dynamics at the phase boundary and cannot be interpreted on the basis of the Burton, Prim and Slichter theory. A transmission micrograph of a GaAs segment cut normal to the growth axis (conventional wafer geometry) and polished on both sides was subjected to dislocation analysis (fig. 5). The visibility of the complex dislocation network is attributed to decoration of the dislocations by dopant elements. It should be pointed out that a comparative analysis of dislocations in GaAs grown by the LEC technique with GaAs grown by the horizontal Bridgman technique revealed fundamental differences in density, distribution and geometry (fig. 6).

Analysis of semi-insulating GaAs indicated transparency, absence of contrast generating media, to 1 μm radiation in bright field transmission mode. Using dark field NIR illumination, extensive scattering centers of submicron dimensions are observed. The scattering centers, precipitates, appear as decorations of dislocations and thus delineate the location of these otherwise invisible defects (fig. 7).

Unambiguous identification of dislocations in semiconductors has thus far been accomplished primarily by electron microscopy and X-ray topography. These approaches are destructive and/or time consuming respectively, thus finding limited application. The third and most widely used approach to dislocation identification, chemical etching, although destructive in nature, is convenient because of its simplicity. Its use is based on the fundamental assumption that the termination of dislocations on the surface gives rise to a local modification of the etch rate and thus results in the generation of etch pits. Etch pit densities are equated with dislocation densities.

There exists in most instances uncertainty as to the one-to-one correspondence between dislocations and etch-pits; chemical etchants can also generate pits that are not related to dislocations and, on the other hand, there is no assurance that all dislocations are revealed through etch pits.

Using near IR bright field transmission microscopy, dislocations in n-type GaAs grown by the horizontal Bridgman technique are revealed in a non-destructive manner through contrast due to impurity decoration of the dislocation lines (fig. 6). By subjecting

the same wafer to photo-etching, etch pits are generated which can be identified (and counted) using interference contrast microscopy with white light in reflection mode (fig. 8a). Operating the microscope in transmission mode, the same sample area yields the transmission image shown in fig. 8b. Accordingly it is established that each etch pit on the surface is associated with a dislocation and, on the other hand, that the termination of each (decorated) dislocation is an etch pit.

It should be pointed out that the establishment of correspondence between dislocations and etch pits cannot be generalized. It must be verified for each particular material system and for the specific etching procedure used.

In the last phase of this research effort commercial GaAs wafers were analyzed for surface damage. It was found that all wafers fail to exhibit processing induced surface and subsurface damage when viewed in reflected NIR Nomarski interference contrast. All wafers, on the other hand, do exhibit varying degrees of subsurface damage when viewed in phase contrast NIR transmission mode (fig. 9).

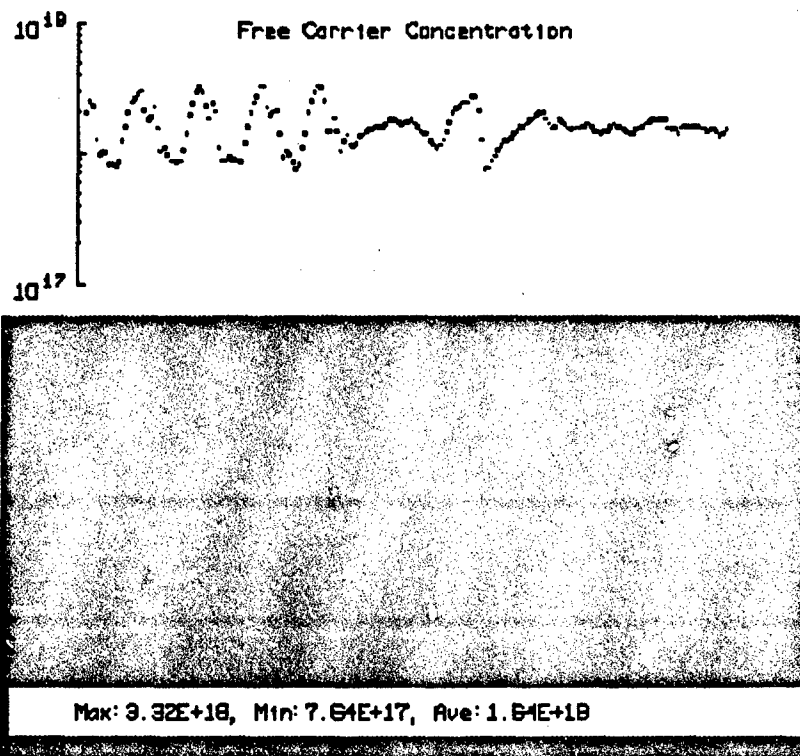


Fig.4 Quantitative Dopant Segregation Analysis in M-LEC GaAs Based on NIR Absorption

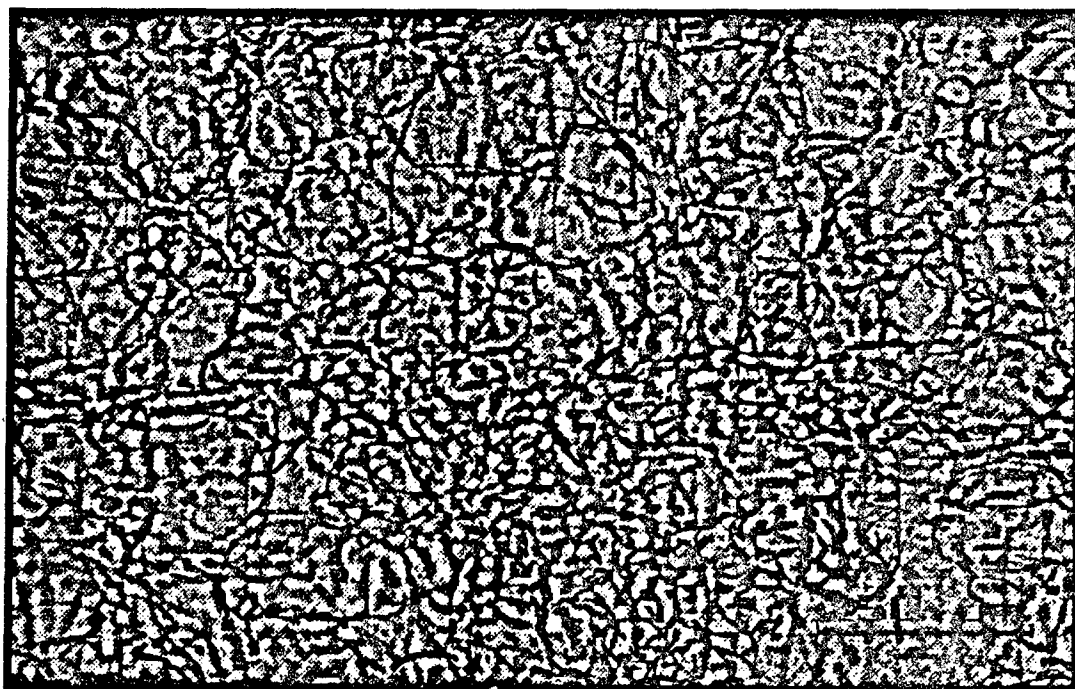


Fig.5 Dislocation Network in Doped LEC GaAs as Revealed by NIR Transmission Microscopy



Fig.6 Dislocations in n-type GaAs grown by the horizontal Bridgman technique, as revealed by NIR transmission microscopy. (Compare with dislocation network in LEC GaAs.)

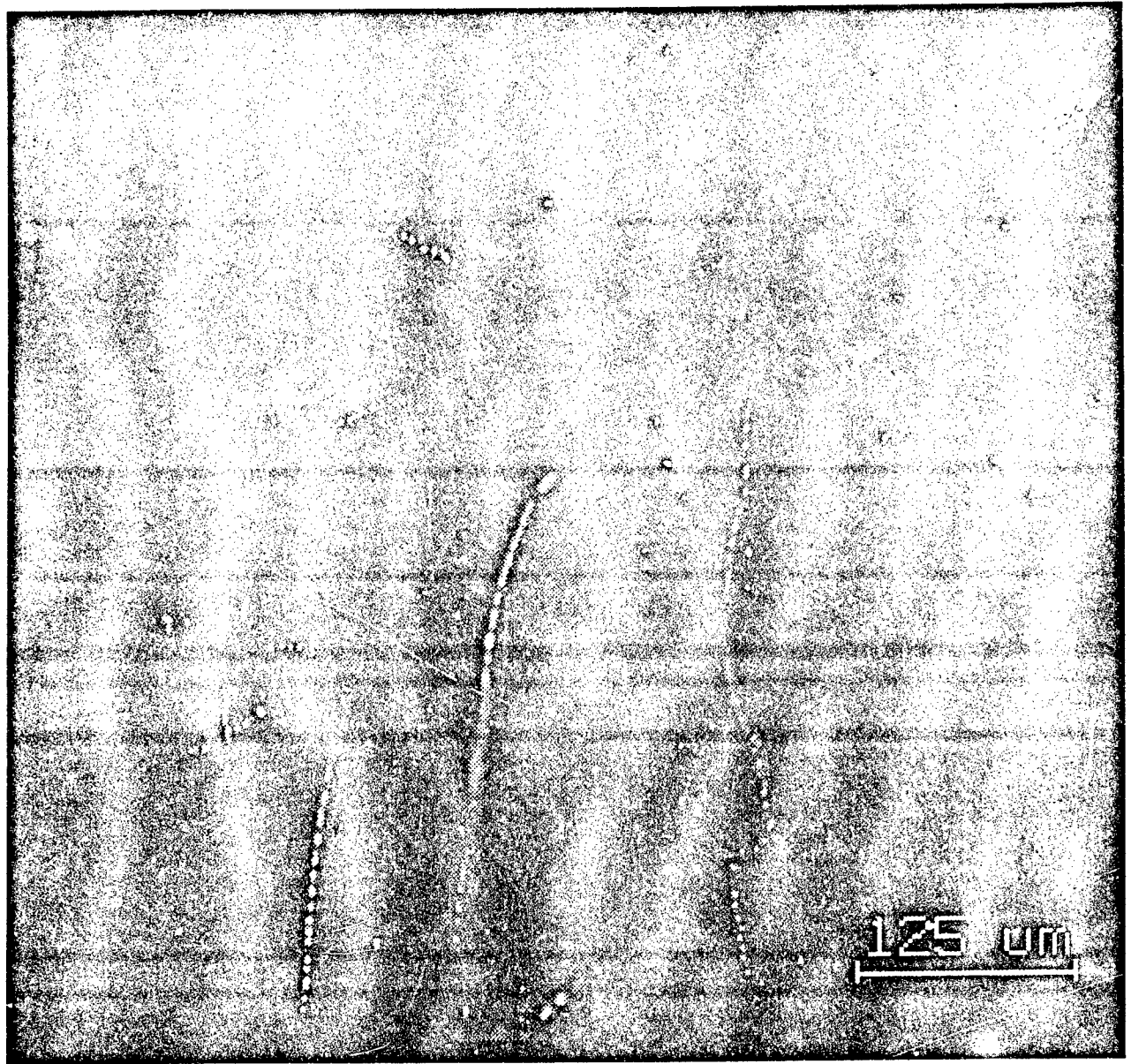


Fig.7 Dislocations as Revealed through Decorating Precipitates in NIR Transmission Darkfield Microscopy.

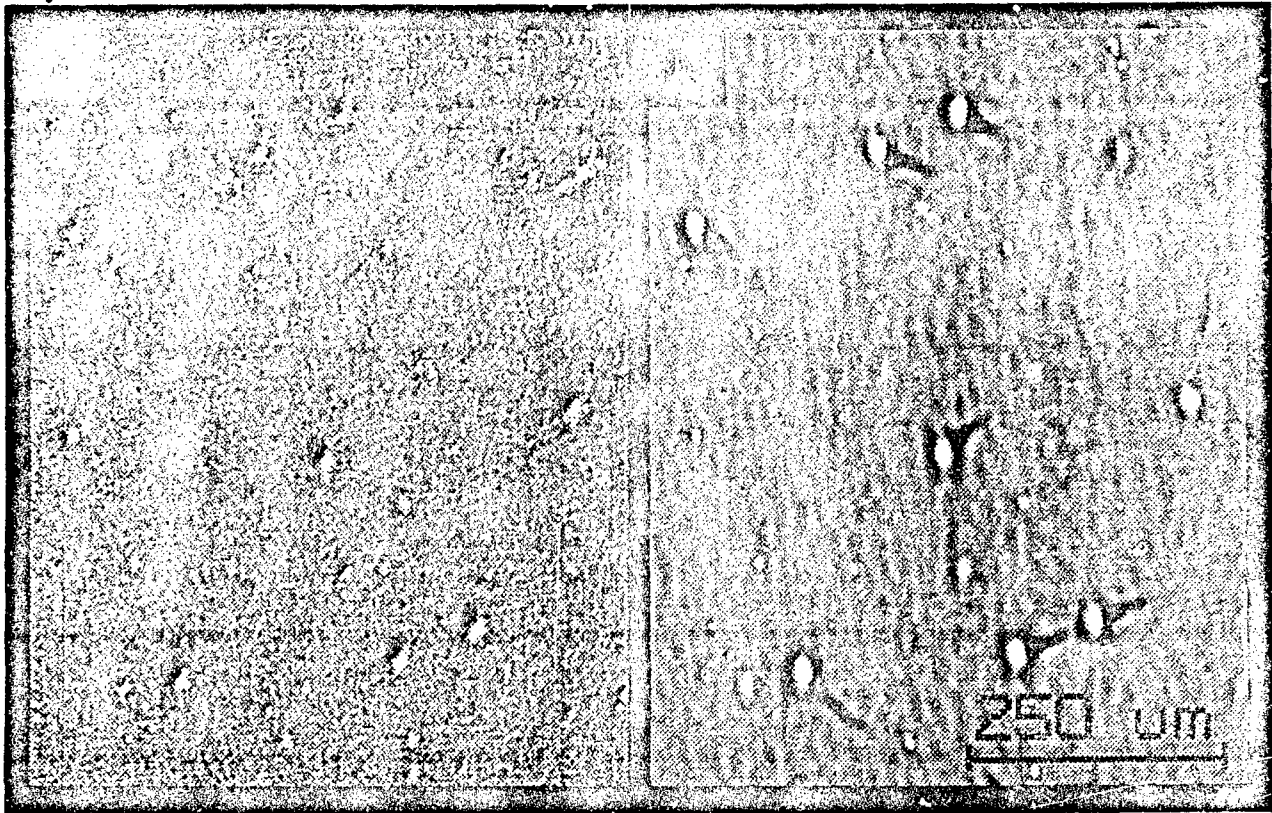


Fig.8 (a) GaAs grown by the horizontal Bridgman technique; the wafer surface shown was subjected to photo-etching; notice etch pits, presumed to correspond to surface terminations of dislocations.
(b) The same area of the wafer as shown in (a) observed in NIR bright field transmission mode; notice the dislocation lines extending from each etch pit.

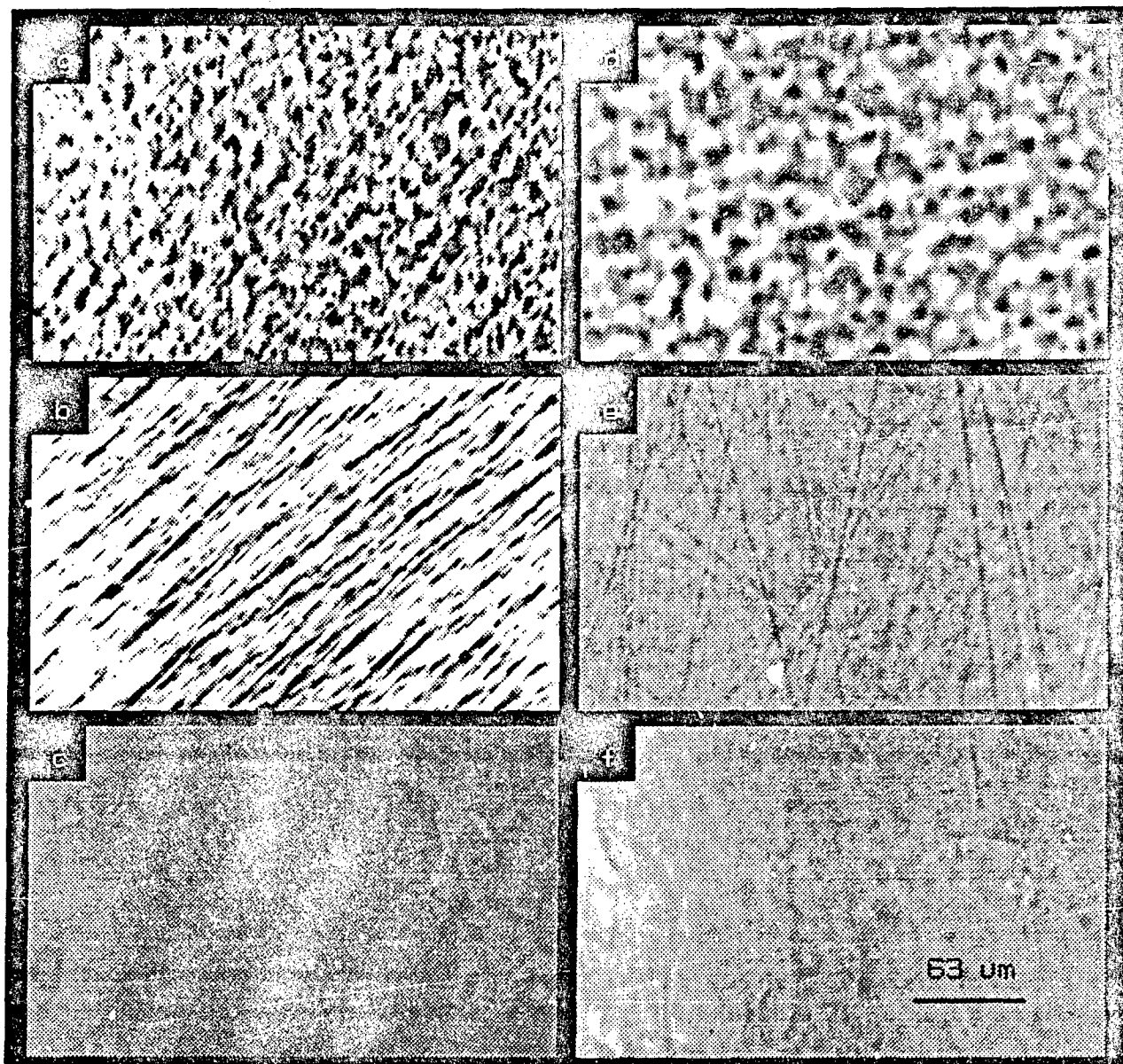


Fig.9 Sub-Surface Damage in Commercial GaAs Wafers as Revealed by NIR Phase Contrast Microscopy in Transmission Mode. (None of the wafers revealed any surface deficiencies when viewed in reflected light Nomarski-type interference contrast microscopy)

CONCLUSIONS

NIR transmission microscopy combined with computational image analysis provides a means for quantitative defect analysis with high sensitivity and spatial resolution. The developed procedures provide micro- and macro-scale analyses which can contribute to an elucidation of growth and segregation phenomena. The sensitivity and resolution achieved permits the identification of gravitational effects on crystal perfection resulting during growth from the melt and from solution. Compatibility of the analytical procedure with tele-operation has been established and is expected to provide for a major advance in efforts to explore the potential of reduced gravity environment for electronic materials research.

PUBLICATIONS

1. D.J. Carlson and A.F. Witt, "Microsegregation in Conventional Si-Doped LEC GaAs", J. Crystal Growth 108 (3/4) 508-518 (1991).
2. X.Z. Cao and A.F. Witt, "Decorated Dislocations in SI-GaAs as Revealed by Dark Field NIR Transmission Microscopy", J. Crystal Growth 112, 838-840 (1991).
3. X.Z. Cao and A.F. Witt, "Identification of Dislocation Etch Pits in n-type GaAs by NIR Transmission Microscopy", J. Crystal Growth 114, 255-257 (1991).
4. D.J. Carlson, M.J. Wargo, X.Z. Cao and A.F. Witt, "New Optical Approaches to the Quantitative Characterization of Crystal Growth, Segregation and Defect Formation", Proceedings of 36th Annual Symposium of International Society for Optical Engineering (1991), p. 140-147.
5. M.J. Wargo and A.F. Witt, "Real Time Thermal Imaging for Analysis and Control of Crystal Growth by the Czochralski Technique", J. Crystal Growth 116 (1/2) 213-224 (1992).

PRESENTATIONS

1. "Emerging Issues in Semiconductors", IAP Course 3.093, MIT, January 24, 1992.
2. "What's New in Semiconductor Growth and Characterization", MESS Seminar, MIT, February 27, 1992.
3. "Non-Invasive Bulk Characterization of GaAs and Si Wafers", Hewlett-Packard, Inc., San Jose, CA, March 4, 1992.
4. "Characterization of GaAs Wafers", Intel Corporation, Santa Clara, CA, March 4, 1992.
5. "Non-Invasive Wafer Characterization", Motorola Corporation, Tempe, AZ, March 6, 1992.
6. "New Approaches to Non-Invasive Semiconductor Characterization", Tenth International Conference on Crystal Growth, San Diego, CA, August 18, 1992.
7. "An Analysis of Status, Problems and Opportunities in Crystal Growth At-large and Controlled Solidification in Particular", EuroCryst Meeting, Vienna, Austria, November 27, 1992.

**END
FILMED**

DATE:

4-93

DTIC