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REPORT SD-TR-89-11

# Subsurface Damage Profiling System for Semiconductor Materials

AD-A206 793

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1 March 1989

Prepared for

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Los Angeles, CA 90009-2960

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This report was submitted by The Aerospace Corporation, El Segundo, CA 90245, under Contract No. F04701-85-C-0086-P00019 with the Space Division, P.O. Box 92960, Los Angeles, CA 90009-2960. It was reviewed and approved for The Aerospace Corporation by S. Feuerstein, Director, Chemistry and Physics Laboratory. Capt John K. Abreu/WCO was the project officer for the Mission-Oriented Investigation and Experimentation (MOIE) Program.

This report has been reviewed by the Public Affairs Office (PAS) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.



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UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE

## REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION Unclassified			1b. RESTRICTIVE MARKINGS		
2a. SECURITY CLASSIFICATION AUTHORITY			3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; distribution unlimited.		
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE					
4. PERFORMING ORGANIZATION REPORT NUMBER(S) TR-0088(3945-09)-1			5. MONITORING ORGANIZATION REPORT NUMBER(S) SD-TR-89-11		
6a. NAME OF PERFORMING ORGANIZATION The Aerospace Corporation Laboratory Operations		6b. OFFICE SYMBOL (if applicable)	7a. NAME OF MONITORING ORGANIZATION Space Division		
6c. ADDRESS (City, State, and ZIP Code) El Segundo, CA 90245			7b. ADDRESS (City, State, and ZIP Code) Los Angeles Air Force Base Los Angeles, CA 90009-2960		
8a. NAME OF FUNDING/SPONSORING ORGANIZATION		8b. OFFICE SYMBOL (if applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER F04701-85-C-0086-P00019		
8c. ADDRESS (City, State, and ZIP Code)			10. SOURCE OF FUNDING NUMBERS		
			PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.
			WORK UNIT ACCESSION NO.		
11. TITLE (Include Security Classification) Subsurface Damage Profiling System for Semiconductor Materials					
12. PERSONAL AUTHOR(S) Ives, Neil A., and Leung, Martin S.					
13a. TYPE OF REPORT		13b. TIME COVERED FROM TO		14. DATE OF REPORT (Year, Month, Day) 1 March 1989	15. PAGE COUNT 20
16. SUPPLEMENTARY NOTATION					
17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)		
FIELD	GROUP	SUB-GROUP	Damage profile, Gallium arsenide, Subsurface damage. (TES)		
19. ABSTRACT (Continue on reverse if necessary and identify by block number)					
Subsurface damage in polished GaAs wafers has been identified by electron microscopy and other surface analytical techniques. Subsurface damage can adversely affect the performance and reliability of devices fabricated on such substrates. In this report, we introduce a method for determining the degree of subsurface damage as a function of depth. Interferometry is used to measure the rate at which substrate material is being etched from the sample. Measuring the etch rate as a function of depth reveals the profile of subsurface damage in the sample. We describe the methodology and present our findings on GaAs wafers that have been subjected to surface damage. Samples obtained from different wafer manufacturers were investigated.					
20. DISTRIBUTION/AVAILABILITY OF ABSTRACT <input type="checkbox"/> UNCLASSIFIED/UNLIMITED <input checked="" type="checkbox"/> SAME AS RPT. <input type="checkbox"/> DTIC USERS			21. ABSTRACT SECURITY CLASSIFICATION Unclassified		
22a. NAME OF RESPONSIBLE INDIVIDUAL			22b. TELEPHONE (Include Area Code)		22c. OFFICE SYMBOL

DD FORM 1473, 84 MAR

83 APR edition may be used until exhausted.  
All other editions are obsolete.SECURITY CLASSIFICATION OF THIS PAGE  
UNCLASSIFIED

PREFACE

We wish to thank A. Pluchino for his valuable advice on interferometry and G. W. Stupian for his helpful comments on the manuscript.

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DTIC TAB	<input checked="" type="checkbox"/>
Unannounced	<input type="checkbox"/>
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CONTENTS

PREFACE..... 1

I. INTRODUCTION..... 7

II. BACKGROUND..... 9

III. THEORETICAL..... 13

IV. EXPERIMENTAL..... 15

V. RESULTS..... 19

REFERENCES..... 25

## FIGURES

1.	Depth (Weight Loss Basis) vs Polish Time for Laminar Flow Polished GaAs Wafer.....	11
2.	Schematic Diagram of Subsurface Damage Profiling System.....	16
3.	Scanning Electron Micrograph of a Fractured Edge from an As-Received GaAs Wafer.....	18
4.	Segment of Raw Data from Depth Profiling Run.....	20
5.	Etch Rate Comparison for Processed GaAs Wafers.....	21
6.	Etch Rate Comparison for GaAs Wafers Supplied by Different Manufacturers.....	22

## TABLE

1.	Profiled Sample Identification.....	17
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## I. INTRODUCTION

Subsurface damage in GaAs wafers, i.e., dislocations and slip planes caused by mechanical stress, may extend as deep as several tens of microns into the substrate. Subsurface damage can adversely affect the performance and reliability of devices fabricated on such substrates. Traditional methods for obtaining detailed measurements of subsurface damage are only sensitive to the first few microns of the surface and do not, in general, provide information about the quality of material that lies beneath the near-surface region.

In this report, we present a method for profiling subsurface damage in GaAs wafers by measuring the etch rate of the wafers as a function of depth, using interferometry. In our technique, the surface of a GaAs wafer acts as the movable mirror in a Michelson interferometer while the wafer is being etched continuously by a laminar flow polishing process (Ref. 1). This polishing process provides optical access to the substrate surface necessary for the interferometric measurement. In contrast to conventional methods for detecting subsurface damage, the technique we have developed has the advantage of being able to monitor damage that lies tens of microns beneath the substrate surface.

## II. BACKGROUND

Subsurface damage in GaAs has been identified by cross-sectional scanning electron microscopy (SEM). The fractured edges of (100) GaAs wafers cleaved along their (011) planes show large crystalline defects on the order of  $0.1 \mu\text{m}$ . Transmission electron microscopy (TEM) has also been used to examine wafers for subsurface damage (Ref. 2). TEM provides very high spatial resolution ( $> 10 \text{ \AA}$ ), but sample preparation is difficult and costly. In addition, sample attrition can be high, especially for a brittle material such as GaAs.

Surface defect delineation by chemical etching can be used to determine the quality of the near-surface region of the GaAs wafer (Refs. 3 and 4). The wafer is exposed to an etching solution that preferentially etches the defects. After etching, the etch pits can be counted using standard optical or electron microscopy. Etch pit counting is useful primarily for determining surface defect densities. If a polishing etch is not used to remove the surface etch pits, they will propagate on subsequent preferential etching, giving an erroneous defect density. For this reason, and because different preferential etch solutions expose different types of defects, the results are difficult to determine unambiguously.

Several commercial systems claim to measure subsurface defects in GaAs wafers. These systems, which rely on optical techniques, are limited to detecting only the optical defects at the surface of the wafer and give little or no information about damage that lies below the characteristic optical penetration depth.

We have developed a simple technique for detecting subsurface damage, based on measuring the etch rate as a function of depth. Damaged material etches more rapidly than damage-free material. In our initial experiments, the sample was etched using a noncontact polishing method developed previously in our laboratory. The sample was weighed at discrete time intervals using an analytical balance. The etch rate was determined as a

function of depth from the measured weight loss as a function of etching time. Samples in which the surface had been abraded showed an etch rate profile much different than that of unabraded wafers. An etch rate profile obtained gravimetrically on a surface scratched by an abrasive is shown in Fig. 1. Material on the surface of the sample was removed at a rate of  $4.4 \mu\text{m}/\text{min}$  for the first  $53 \mu\text{m}$  and  $1.3 \mu\text{m}/\text{min}$  for the next  $80 \mu\text{m}$ . These results show that the near-surface region has more damage than the bulk substrate. The gravimetric method for studying damage profiles is simple and direct but lacks depth resolution. The tedium of this method, which requires many repetitive weighings, led us to develop a method for measuring etch rate profiles in real time using interferometry.

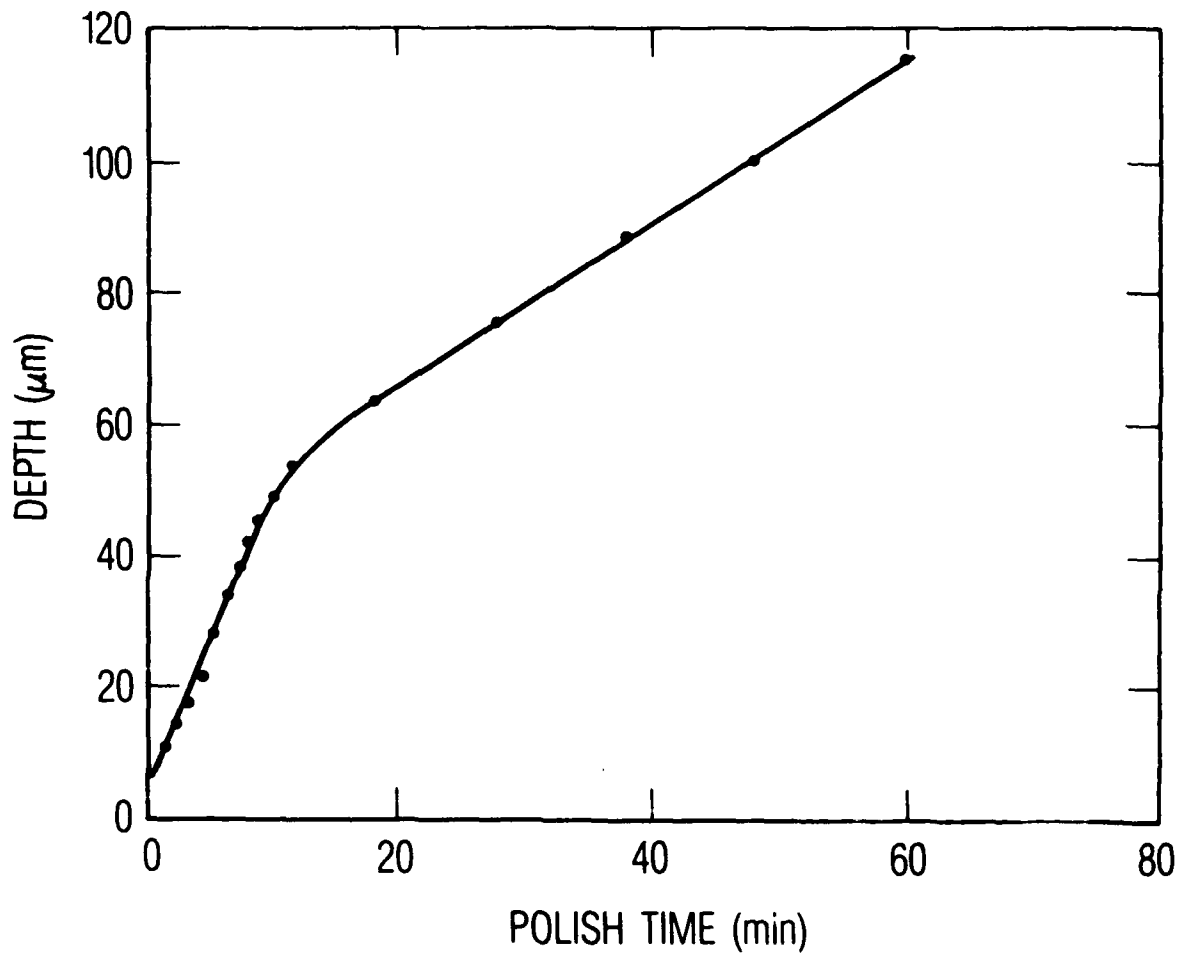


Fig. 1. Depth (Weight Loss Basis) vs Polish Time for Laminar Flow Polished GaAs Wafer

### III. THEORETICAL

Using the GaAs wafer surface as one of the mirrors in a Michelson interferometer, a pattern of interference fringes can be obtained. As material is removed from the surface of the wafer, the relative distance between the mirrors increases, causing the interference fringes to shift. The rate at which the fringes shift is a measure of the rate at which material is removed from the wafer.

A simple analysis (Ref. 5) shows that the change in sample thickness can be expressed as

$$d_0 - d_t = (m_0 - m_t)\lambda/2n_s$$

where  $d_0$  is the thickness at  $t = 0$ ,  $d_t$  is the thickness at time  $t$ ,  $m_0$  is the fringe order corresponding to  $d_0$ ,  $m_t$  is the fringe order corresponding to  $d_t$ ,  $\lambda$  is the wavelength of monochromatic light used for the measurement, and  $n_s$  is the refractive index of the etch solution.

By continuously monitoring the number of fringes in the interference pattern that sweep by a fixed point, we can determine the etch rate as a function of time and depth.

#### IV. EXPERIMENTAL

The experimental apparatus is shown in schematic form in Fig. 2. The main optical parts of the system are a highly reflective plane mirror (M), the reflective GaAs substrate (S) mounted in the polishing optical flow cell, and the antireflection coated beamsplitter (T). The light emitted from the HeNe laser source (L) is divided by a beamsplitter into a reflected beam and a transmitted beam of approximately equal intensity. The reflected beam arrives at the photodiode via the plane mirror and the beamsplitter. The transmitted beam is reflected by the substrate surface and the beamsplitter onto the photodiode, where it is recombined with the reflected beam. To obtain fringes, the mirror and the sample are placed perpendicular to each other by micrometer adjustments on the mirror. A 1 mm aperture is placed in front of the photodiode to resolve the fringes in the interference pattern. The etching fluid (0.5% Br in methanol) is pumped up to an elevated reservoir and gravity fed to the optical cell to decouple the mechanical vibration from the recirculation pump.

The as-received samples used in the preliminary study were (100) GaAs wafers with defect densities on the order of  $10^4/\text{cm}^2$ . The wafers were cut into sections, and each section was given a different surface treatment. One of the sections was cleaned by rubbing the surface lightly with a cotton tip swab, which is a typical wafer cleaning process used in the industry. Another section received 60 min of laminar flow polishing, which has been shown to remove subsurface damage effectively.

Diverse wafer samples were obtained from several manufacturers. These wafers represented defect densities ranging from  $\geq 3 \times 10^4/\text{cm}^2$  to  $\leq 3 \times 10^2/\text{cm}^2$ . Information concerning each sample is given in Table 1. The wafers were packaged in polypropylene shipping trays and polypropylene paper envelopes isolated by foam inserts. Each sample was profiled for 30 min. A scanning electron micrograph showing the subsurface of a typical fractured edge of an as-received sample with subsurface damage is shown in Fig. 3.

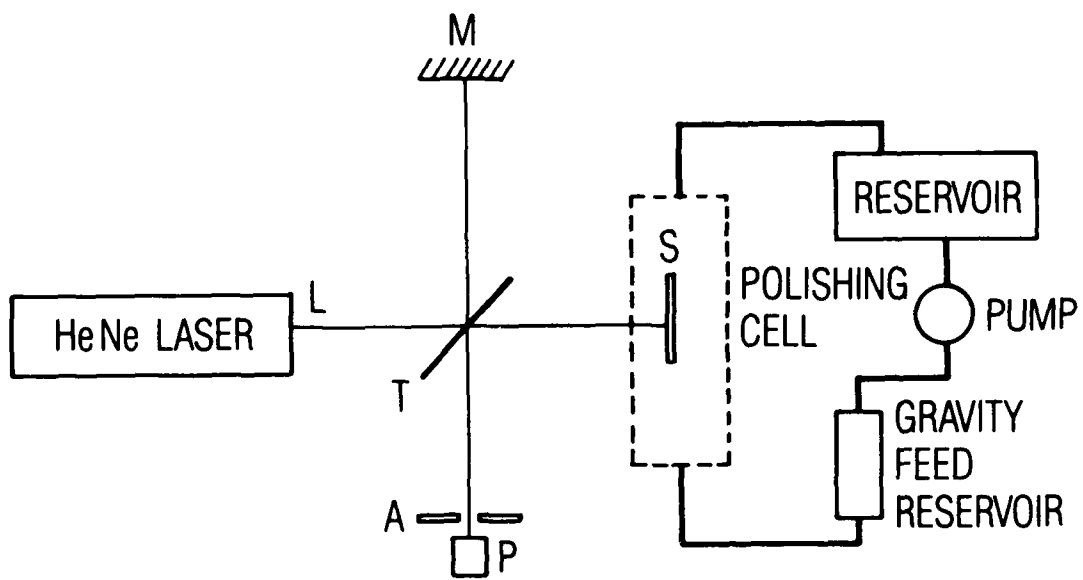


Fig. 2. Schematic Diagram of Subsurface Damage Profiling System

Table 1. Profiled Sample Identification

Profile No.	Manufacturer	Defects/cm <sup>2</sup>	Packaging	Miscellaneous Notes
1	A	<10 <sup>4</sup>	PPWC <sup>a</sup>	Handling with tweezers
2	B	<10 <sup>4</sup>	PPWC <sup>a</sup>	Handling with tweezers
3	A	<10 <sup>4</sup>	PPWC <sup>a</sup>	Postlaminar flow polish (60 min)
4	C	>300	PP-P <sup>b</sup>	As received
5	D	>300	PPWC <sup>a</sup>	Lightly scratched, handling with tweezers, poor packing

<sup>a</sup>Polypropylene wafer carrier

<sup>b</sup>Polypropylene paper envelope

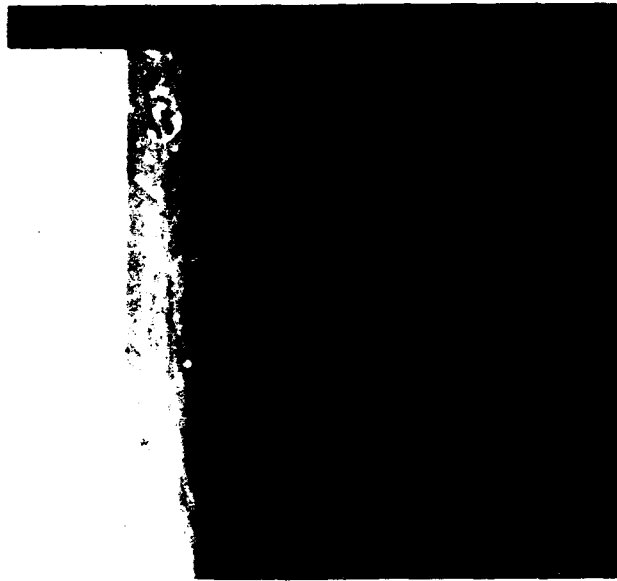


Fig. 3. Scanning Electron Micrograph of a Fractured Edge from an As-Received GaAs Wafer

## V. RESULTS

Preliminary results show that we are able to measure etch rates as a function of depth in GaAs samples. These results confirm the presence of a region of subsurface damage in the as-received samples. A plot of the amplitude of the light intensity detected as the interference fringes shift during etching is shown in Fig. 4. By counting fringes, we directly determine the change in thickness of the wafer as a function of time. The rate of change of thickness, i.e., the etch rate, is an indicator of the extent of subsurface damage in the wafer.

Etch rates determined for three samples subjected to different handling and treatment are shown in Fig. 5. Sample 1, which was deliberately rubbed with a cotton swab, exhibits a considerably higher etch rate than does an as-received wafer (sample 2), indicating a higher degree of subsurface damage after rubbing. After laminar flow polishing (sample 3), the etch rate of the as-received sample is lowered, consistent with the behavior expected after removal of a damaged surface layer.

Etch rate profiles for wafers received from different manufacturers are shown in Fig. 6. The profiles show that the material etches more rapidly at the surface than in the bulk. The etch rate of sample 1 remains relatively high as the first 15  $\mu\text{m}$  of the material is removed. We find that a layer of subsurface damage 15  $\mu\text{m}$  thick is typical of wafer surfaces prepared by a combination of chemical and mechanical polishing. The etching profile of sample 2 is similar in shape to that of sample 1, but the region of subsurface damage extends much deeper into the bulk material. Profile 3 illustrates the improvement in sample quality after 60 min of laminar flow polishing. The lower removal rate is consistent with material of low subsurface damage. Sample 4 was by far the best specimen studied. It had the least amount of subsurface damage and good crystal quality, as evidenced by a low bulk etch rate. A low bulk etch rate, after removal of surface layers, implies low defect density and thus provides an assessment

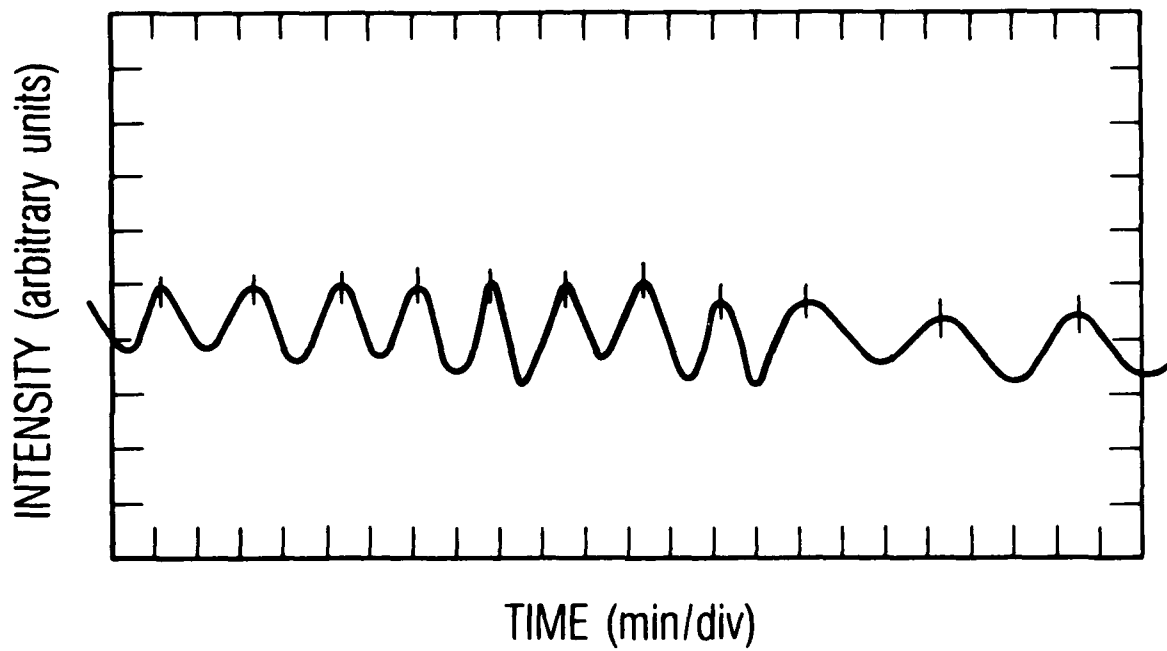


Fig. 4. Segment of Raw Data from Depth Profiling Run

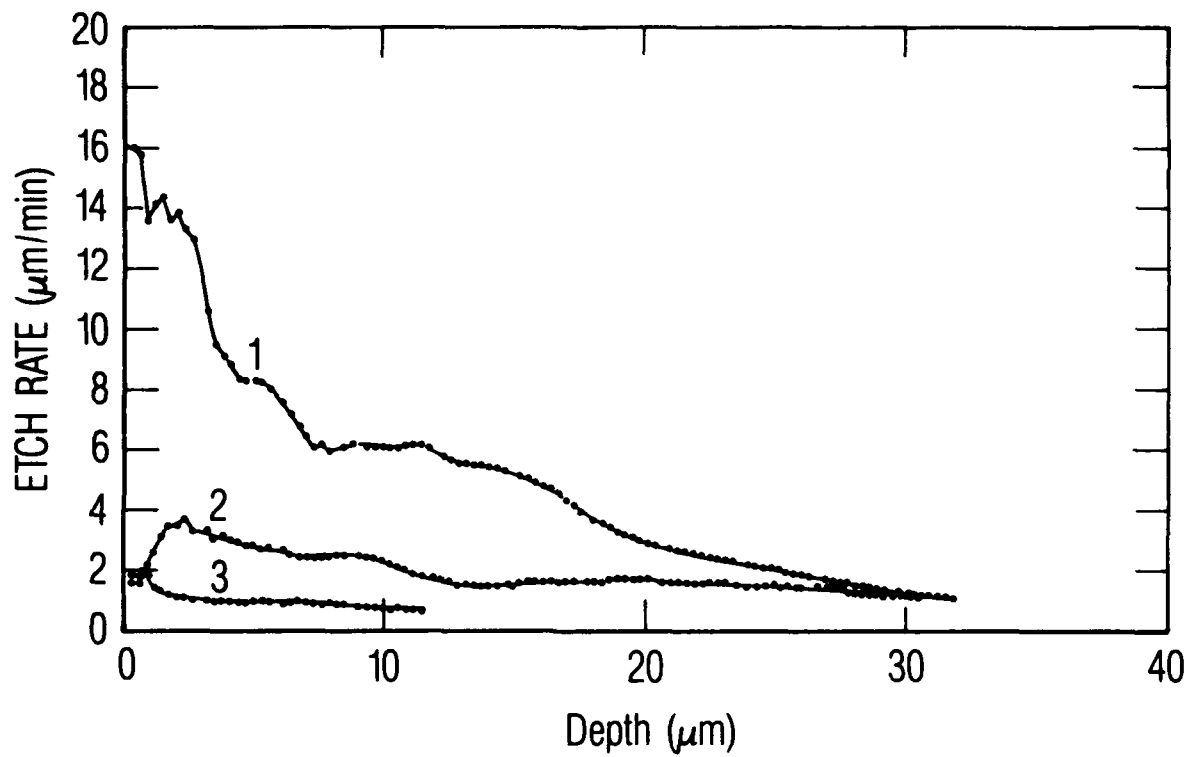


Fig. 5. Etch Rate Comparison for Processed GaAs Wafers. (Refer to text for identification.)

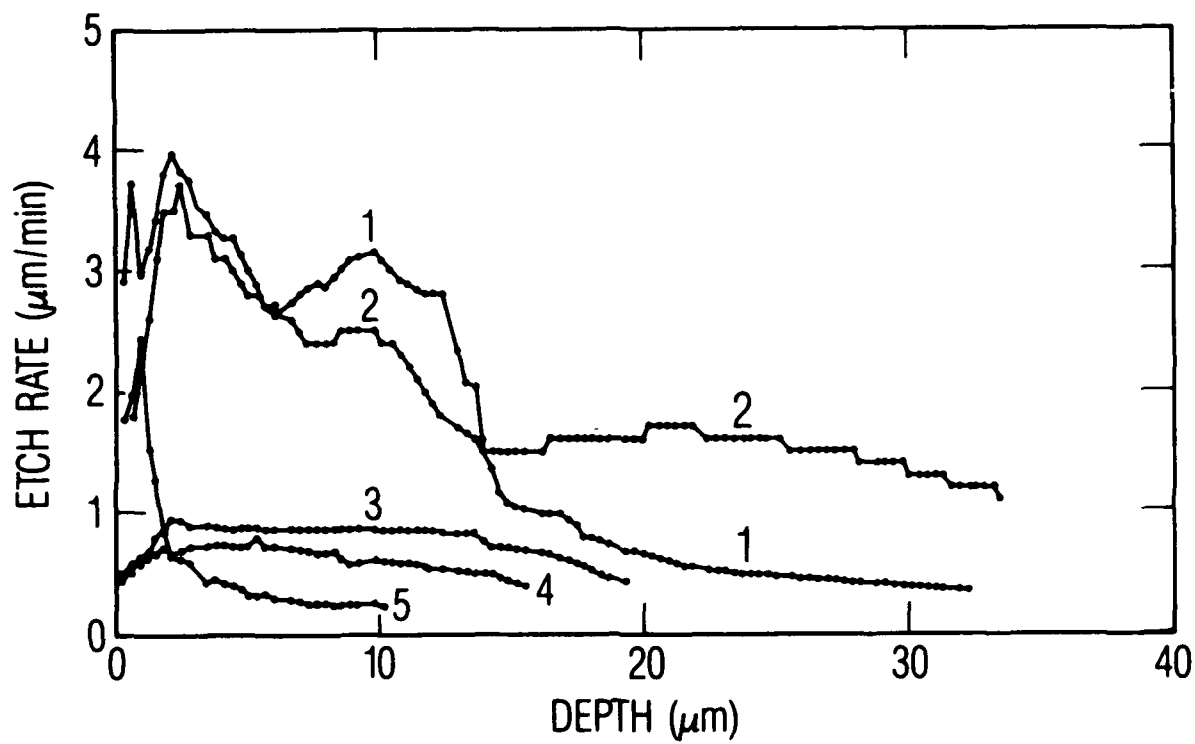


Fig. 6. Etch Rate Comparison for GaAs Wafers Supplied by Different Manufacturers. (Refer to text for identification.)

of "crystal quality." Sample 5, which was an indium stabilized GaAs wafer, had the lowest bulk etch rate of all specimens examined. Addition of a few percent of indium is believed to aid in the reduction of dislocations within the bulk material (Ref. 6). When initially examined, the wafer had a significant amount of visible surface damage, but this damage extended only 4  $\mu\text{m}$  into the material. This result suggests that the indium stabilized wafer had superior crystalline quality to the other wafers examined.

The GaAs wafers were etched preferentially by photochemical reactions in areas illuminated by the laser beam. These areas developed a slight concave "crater" that extended into the bulk. There is a correlation between laser light intensity and the depth of the craters formed during the profiling. Because the light intensity of the laser and the concentration of the solution were kept constant, the effect should be the same for all samples evaluated.

Our results indicate that we are able to characterize subsurface damage and evaluate crystalline quality in a number of different wafers by measuring the etch rate vs depth profiles. Evidence of subsurface damage was found in most of the as-received samples. The ability to characterize the extent of subsurface damage is critically important to understanding how to improve device performance and reliability. Our method provides a practical means for determining the quality of GaAs wafers for device fabrication. With the proper choice of etching solution, the method can be modified to evaluate other semiconductor materials.

## REFERENCES

1. N. A. Ives and M. S. Leung, Rev. Sci. Instr. 59, 172 (1988).
2. D. K. Sadana, M. Stratham, J. Washburn, and G. R. Booker, J. Appl. Phys. 51, 175 (1975).
3. W. C. Niehaus and B. Schwartz, Solid State Electron. 112, 175 (1975).
4. T. S. Plasett and A. H. Parsons, J. Electrochem. Soc. 112, 954 (1965).
5. F. A. Jenkins and H. E. White, Fundamentals of Optics, 4th ed., McGraw-Hill Book Company, Inc. (1979), p. 282.
6. Y. Seki, H. Watanabe, and J. Matsui, J. Appl. Phys. 49, 822 (1978).

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