

Deposition Parameter Studies and Surface Acoustic Wave Characterization of PECVD Silicon Nitride Films on Lithium Niobate

Jacqueline H. Hines, *Member, IEEE*, Donald C. Malocha, *Fellow, IEEE*,
Kalpathy B. Sundaram, *Senior Member, IEEE*, Kevin J. Casey, and Kwang R. Lee

Abstract—Silicon nitride films were deposited by a plasma enhanced chemical vapor deposition technique using silane-ammonia as the reactant gas mixture. The influence of the process parameters such as flow ratio of the reactant gases, pressure, substrate temperature, RF power, time of deposition and electrode spacing on the deposition and etch rates were investigated. From the matrix of deposition conditions, the deposition parameters for high quality films applicable to surface acoustic wave (SAW) technology were found. Experimental results on the acoustic loss, reflectivity and velocity dispersion for the fabricated devices are presented.

I. INTRODUCTION

SILICON nitride (Si_3N_4) prepared by low temperature plasma enhanced chemical vapor deposition (PECVD) is one of a group of thin film dielectrics which has found wide application in semiconductor devices and circuits, and in the integration of acoustic wave devices on semiconducting substrates [1]–[3]. Such dielectric thin films can also be useful when incorporated into SAW devices on traditional piezoelectric substrates such as lithium niobate (LiNbO_3). When properly configured, such layers can be used to provide various functions including device passivation, waveguiding, and reflective structures.

The acoustic velocity of amorphous Si_3N_4 formed by PECVD is determined by the film density and the compressional and shear elastic constants c_{11} and c_{44} . These quantities vary with the PECVD deposition parameters: substrate temperature, pressure, RF power, the flow ratios of the reactant gases and the electrode spacing (in the case of a parallel plate system). All of these parameters substantially affect the deposition and etch rates and other physical and optical properties of the film. In previous work [4]–[6], a deposition parameter matrix was constructed for a SiH_4 – NH_3 PECVD process, and the effects of variation of the above parameters on the deposition and etch rates were

studied. A set of deposition parameters which result in high quality films applicable to SAW devices were determined. The acoustic properties of Si_3N_4 deposited on LiNbO_3 under these conditions were investigated using surface wave devices fabricated using silicon nitride film pads and reflective strip arrays between two unweighted transducers. Acoustic properties such as propagation loss, velocity dispersion, and reflectivity were studied.

II. FILM DEPOSITION CONDITIONS

The silicon nitride films were grown in a modified Plasma Technology reactive ion etch (RIE) system (Model RIE-80). With a few hardware changes, this system can be reconfigured for plasma enhanced chemical vapor deposition (PECVD). When configured for PECVD, the system is a parallel plate reactor with a mechanical pump for roughing and a molecular drag pump able to provide a background pressure of 2×10^{-5} Torr in the chamber prior to flowing the process gases. The water cooled upper electrode plate is 24 cm in diameter and uses a “shower head” assembly with 17 holes within a diameter of 7.6 cm to uniformly distribute the process gases. The lower plate is 24 cm in diameter, with a 95% uniformity diameter of approximately 15 cm, and can be water cooled or heated with the substrate heater to 400°C. The RF power (13.56 MHz) is connected to the top plate. Films were grown using combinations of silane and ammonia. A 3% silane in helium mixture was used as the silicon reactant source.

The substrates used for deposition characterization studies were *n*-type silicon (100) wafers. After obtaining the initial high vacuum, the throttling was adjusted to obtain the required pressure for deposition. The system was then purged with ammonia at this pressure so as to clean the system from any residual oxygen. Before introducing the silane, the plasma was established with ammonia and this plasma was continued for two to three minutes. The required deposition parameters were then adjusted and all the conditions were stabilized before the deposition was started.

The deposition and etch rates of the films were studied with respect to variations in the deposition parameters: flow ratio of silane to ammonia, chamber pressure, RF power, substrate temperature, deposition time, and electrode spacing. The range of these parameter variations performed are listed in

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J. H. Hines, D. C. Malocha, K. B. Sundaram, and K. J. Casey are with the Department of Electrical and Computer Engineering, University of Central Florida, Orlando, FL 32816-2450 USA.

K. R. Lee is with the Electronic Signal Processing Laboratory, Institute of Advanced Engineering, C.P.O. Box 2810, Seoul 110-714, Korea.

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TABLE I
RANGE OF DEPOSITION PARAMETERS EVALUATED

Parameter	Range of Values			
Silane/Ammonia Flow Ratio	0.01	0.05	0.10	1.00
Chamber Pressure, mTorr	100	200	500	1000
R.F. Power, Watts	10	50	100	150
Substrate Temperature, °C	30	100	200	350
Time, minutes	5	10	20	30
Electrode Spacing, mm (ES)	30	40	50	60

Table I. The etch rates were determined by etching the silicon nitride film with buffered oxide etch (13 NH_4F :2 HF) 9 : 1 diluted in water. Following optimization of film deposition parameters for acoustic device applications, film compositions were studied using Auger spectroscopic analysis.

III. EVALUATION OF FILM DEPOSITION RESULTS

A. Effect of Silane to Ammonia Flow Ratio

The range of silane to ammonia flow ratio tested was from 0.01 to 1.0. Fig. 1 shows the variation of deposition rate with respect to an increasing gas flow ratio of SiH_4/NH_3 for two pressures (300 and 700 mTorr). The RF power and the substrate temperature were kept constant. It is seen that the deposition rate slowly decreased when the flow ratio increased. The deposition rates have similar values at a flow ratio of 0.5 as reported by Sinha *et al.* [7] and the deposition rate is higher at higher chamber pressure conditions. Several studies at different deposition conditions showed a similar trend for the deposition rate with increase in the flow ratio. This is contrary to the results reported by Sinha *et al.* and Piccirillo *et al.* where it is reported that as the flow ratio increases the deposition rate also increases [7]–[8]. However, Piccirillo's measurements of deposition rate versus flow ratio were performed at an RF power of 20 Watts, well below the power at which minimum deposition rate occurs. Osenbach *et al.* while examining the flow ratio range between 0.2–2.0 found that the deposition rate increased initially, reaching a maximum value and finally showed a decreasing trend [9]. In the present work, very small flow ratio experiments could not be performed because of the limitation of the flow meters and, for the given deposition conditions, the deposition rate showed a decreasing trend for an increase in flow ratio.

Fig. 2 shows the plots of the wet etch rate versus the flow ratio of silane to ammonia for films deposited with two different electrode spacings. Similar results were reported by other workers showing that the increase in flow ratio causes a decrease in etch rate [7], [8], [10], [11]. This is due to the fact that the films deposited under high silane condition have more silicon and less hydrogen and nitrogen content in the film

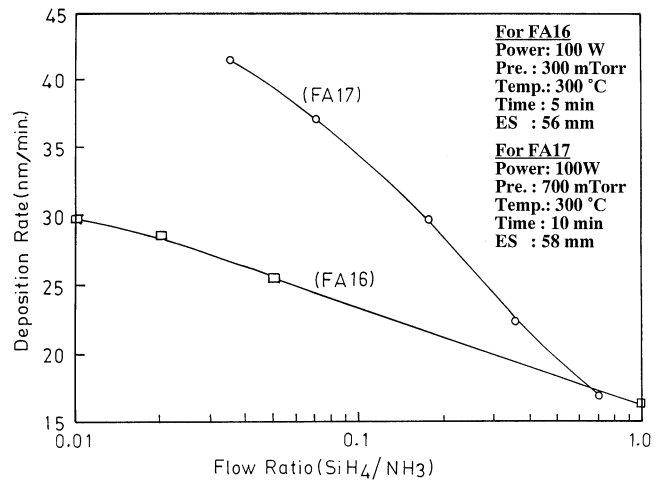


Fig. 1. Deposition rate versus SiH_4/NH_3 flow ratio.

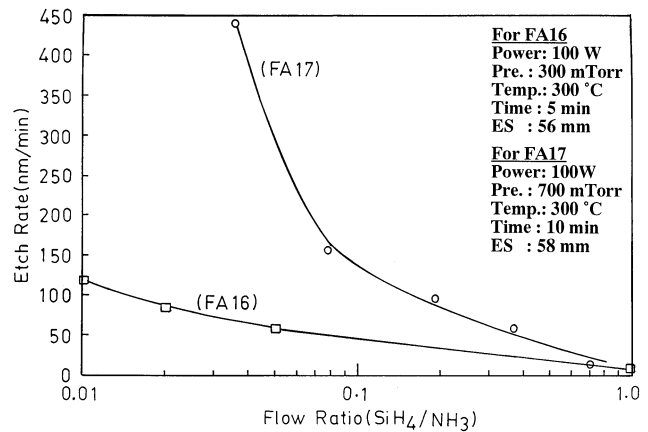


Fig. 2. Etch rate versus SiH_4/NH_3 flow ratio.

which decreases the etch rate [12]. The low etch rate observed at high flow ratios is also indicative of high density films.

B. Effect of Pressure

The chamber pressure was varied from 200 to 1000 mTorr during deposition. Increase in the chamber pressure caused both deposition and etch rates to increase as shown in Fig. 3. The increase in pressure causes more ionized radicals in the processing ambient causing an increase in the deposition rate. The increase in deposition rate will, in turn, correspond to less dense films which can be etched faster, as observed by Sinha *et al.* and Hey *et al.* [7], [13]. As the density of the films decrease, acoustic losses tend to increase due to scattering caused by voids.

C. Effect of Power

The RF power was varied in the range of 10 to 200 Watts for depositions which corresponds to a power density of 22–440 mWatt/cm^2 . The increase in RF power caused increases in both deposition and etch rate as shown in Fig. 4. Similar results were reported by other workers [14], [15]. Sinha *et al.* found no critical change in density and etch rate with respect to increase

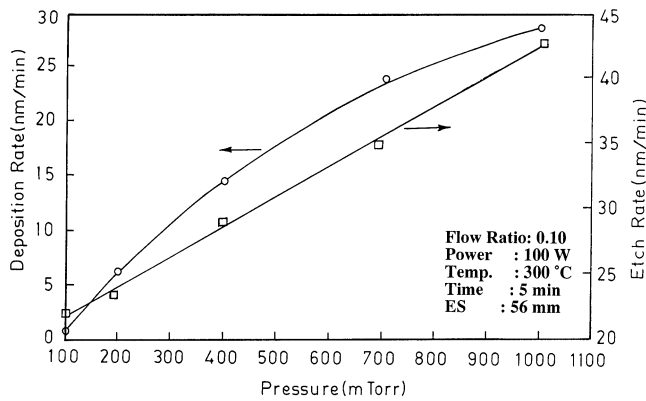


Fig. 3. Deposition and etch rates versus chamber pressure.

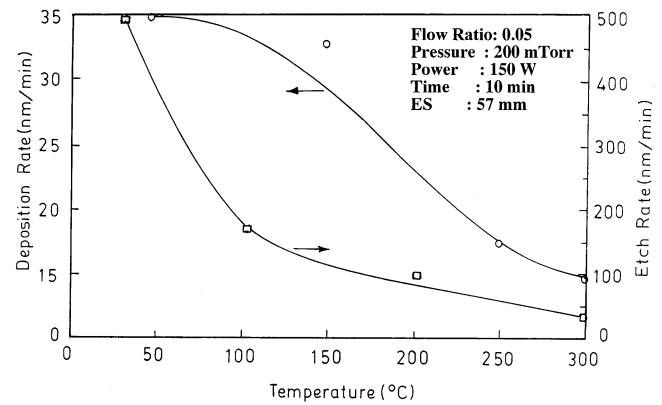


Fig. 5. Deposition and etch rates versus temperature.

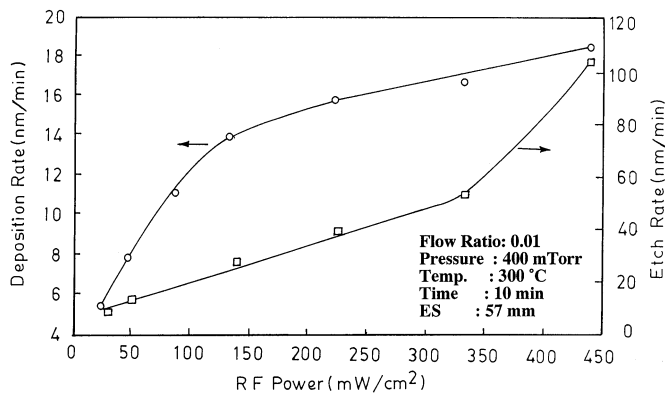


Fig. 4. Deposition and etch rates versus RF power.

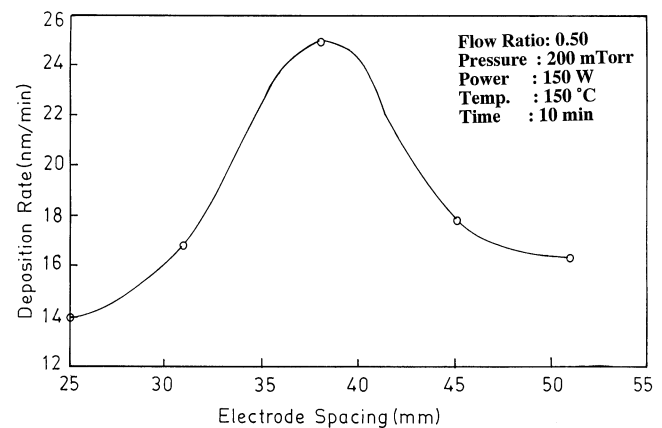


Fig. 6. Deposition rate versus electrode spacing.

in RF power, though deposition rate increased [7]. Piccirillo *et al.* observed a deposition rate with a strong RF power dependence which exhibited a minimum of slightly under 100 Å/min near 50 watts for a wide range of flow ratios. The deposition rate was observed to increase to over 200 Å/min for both higher and lower power levels. Piccirillo also observed a corresponding minimum in the etch rate, corresponding to the decrease in density with increasing film deposition rate [13]. This minimum is not observed in the current work since the lowest power considered is approximately 50 Watts. Also, a slightly higher maximum power (200 Watts) is considered here, while Piccirillo maintained power at or below 150 Watts.

D. Effect of Substrate Temperature

The effect of temperature on the deposited film was investigated by varying the temperature from room temperature to 350°C, keeping the other parameters fixed. Fig. 5 shows the effect of temperature on deposition rate. As temperature increased the deposition rate decreased. Roster *et al.* [16] concluded that accurate temperature control is not very important in a CVD process activated not by temperature but by an RF glow discharge because it is found that the deposition rate decreased by only 3% for every 50°C increase in temperature [8]. This is contrary to the current findings, where it is observed that deposition rate can decrease by up to 25% for

a 50°C increase in temperature. Films produced at higher temperature will tend to have less hydrogen content, be more dense, and etch more slowly, all desirable film properties for SAW applications. Fig. 5 also shows this effect of deposition temperature on etch rate, and similar results have been reported by other researchers [7], [8], [11].

E. Effect of Electrode Spacing

The electrode spacing was varied between 25 mm and 58 mm for the silane-ammonia process. The behavior of the deposition rate is shown in Fig. 6. The electrode spacing to achieve highest deposition rate for the stated deposition parameters is approximately 38 to 40 mm. Since the density of the deposited film is inversely related to deposition rate, the electrode spacing to achieve high quality films for acoustic applications under optimum SAW film deposition conditions was selected as 57 mm.

F. Optimum SAW Film Deposition Conditions

Silicon nitride films which are desirable for SAW device applications have densities nearing those of bulk Si₃N₄, indicating high quality films with few voids. Such films result in high acoustic reflectivity, a large change in acoustic velocity due to stiffening of the surface of the piezoelectric substrate,

and inherently low propagation loss under the film relative to other dielectrics. These effects allow for implementation of acoustic reflecting structures and waveguiding structures with thin nitride film thickness', further reducing acoustic losses due to presence of the film. In order to obtain dense, high quality films, it is necessary to deposit the films under conditions which limit the amount of hydrogen incorporated into the films, and allow for film growth at low deposition rates. Deposition conditions which yielded excellent film properties for use in the acoustic devices under consideration were chosen from the matrix of depositions described above. System operation was optimized with the following deposition parameters:

Power Density = 663 mW/cm² RF Power = 100 Watts
 Electrode Spacing = 57 mm Pressure = 700 mTorr
 Gas Flow = SiH₄/NH₃ = 0.705 Temperature = 300°C

Films were deposited with deposition times ranging from 6 minutes to 30 minutes. The deposition rate with the conditions specified above was approximately 110 Å/min.

G. Analytic Film Studies

Films deposited under the optimized conditions described above were observed to have a wet etch rate of 22 ± 7 Å/min in buffered oxide etch at room temperature. This slow etch rate is due to the high density of the films, measured to be 2.96 ± 0.07 g/cm³, fairly close to the density of bulk Si₃N₄, 3.1 g/cm³. The slight decrease in density with increasing film thickness observed by Hickernell [3] was not seen for films with thickness' between 800 Å and 3600 Å. It is well documented that silicon nitride films fabricated by low temperature PECVD using silane and ammonia are often high in hydrogen content [3], [8], [18], [19], resulting in variation in physical and chemical film properties. High densities imply low hydrogen content and stoichiometry nearing Si₃N₄. The density reported by Taylor for a film with the composition Si₃N₄ was 3.00 g/cm³ [19]. Interpolating Taylor's measured densities and stoichiometries, the measured densities for the films produced in the current work indicate that these films are approximately Si₃N_{3.898}. The compositions of the films were analyzed using Auger electron spectroscopy (AES). The AES depth profiling of the films was done to a depth of 1500 Å by sputter etching the film and making a measurement every 100 Å. A typical AES depth profiling of the silicon nitride film is shown in Fig. 7 indicating that the composition of the film was fairly uniform, with very low carbon and oxygen contents. An estimation of the average composition of the film based on AES spectra was found to be Si₃N_{3.886}O_{0.05}, in good agreement with the density-based estimate. Infra-red spectroscopic analysis confirmed the low hydrogen content of these films.

IV. SAW DEVICE DESIGN AND RESULTS

Simple delay lines were fabricated on 128° YX and YZ LiNbO₃ for measurement of the SAW properties. Three different fundamental frequency devices were designed having

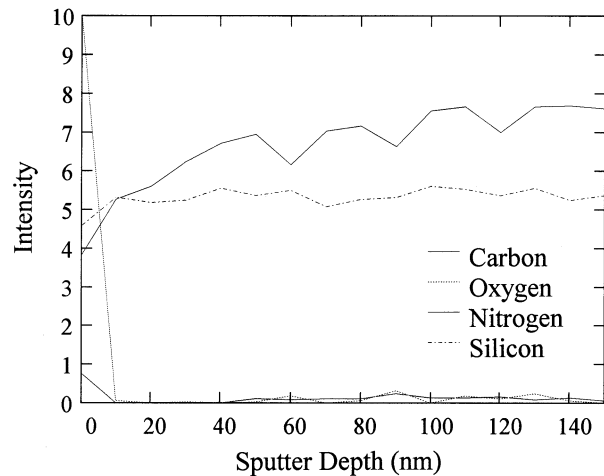


Fig. 7. AES depth profile of silicon nitride films.

wavelengths of 24 μm, 40 μm, and 80 μm. All devices used split electrodes to eliminate internal transducer reflections. The transducers were 10λ long with seven dummy electrodes at each end and acoustic beam widths of 50λ. The devices generated both fundamental and harmonic responses, and measurements were made at the fundamental and all harmonic frequencies that had good suppression of RF feedthrough relative to the SAW delay line response. This approach provided multiple data points of normalized thickness without the need for design and fabrication of a large number of devices. Control devices were fabricated without the presence of silicon nitride thin films. Two port *S*-parameter data was obtained from the control device at the fundamental and harmonic frequencies of interest. Through parameter extraction techniques, the transducer electrode thin film loss, electrical mismatch loss, and propagation loss were found at every frequency of interest. This technique of parameter extraction takes into account the effects of varying electrode duty cycle and small fabrication defects which affect thin film loss and electrical mismatch loss as a function of frequency [17].

Sets of test devices were then fabricated with a silicon nitride film pad in the propagation path between the two transducers. The film extended well beyond the edges of the SAW aperture to eliminate waveguiding effects. The effective propagation length of the SAW under the silicon nitride film was approximately 10 mm and the transverse ends of the film were slanted to eliminate acoustic reflection effects. Device measurements were made at each frequency of interest. Parameter extraction was then conducted to eliminate the thin film and electrical mismatch losses for each transducer. The propagation loss from the control devices was then subtracted, leaving only the propagation loss due to the silicon nitride film. Velocity measurements were made by measuring the group delay of the *S*₂₁ data at the frequencies of interest. The velocity data was corrected for free surface regions and for the regions under the transducer.

Fig. 8 shows the linear characteristic of measured SAW propagation loss versus normalized film thickness. The

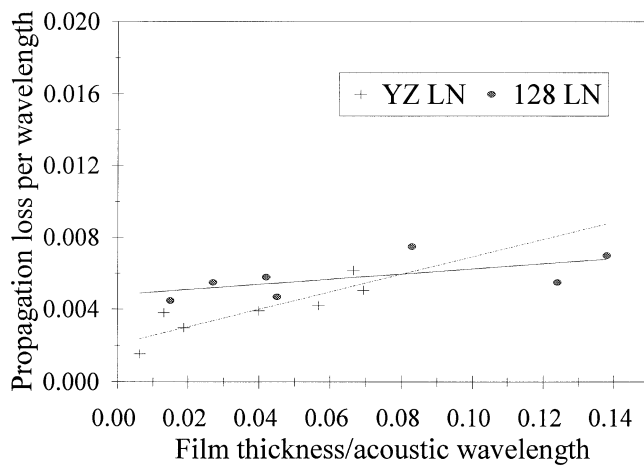


Fig. 8. SAW propagation loss versus normalized film thickness.

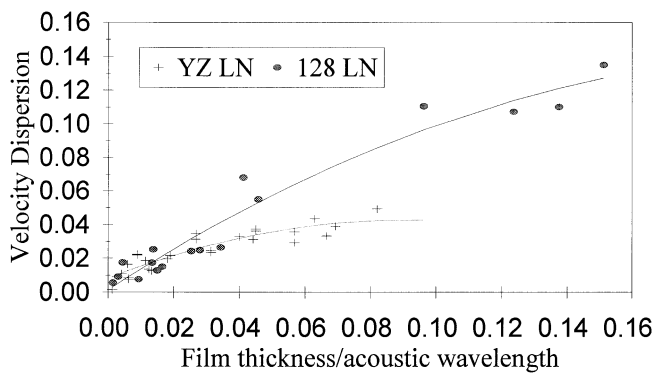


Fig. 9. SAW velocity on 128° YX and YZ LiNbO₃ versus normalized film thickness.

PECVD films selected for use in acoustic devices show propagation loss much lower than the 0.02 dB/ λ reported for SiO₂ sputtered films of equivalent normalized thickness on LiNbO₃ [4], [5], [20]. It is believed that the low propagation loss observed is a result of high quality films with few voids.

The SAW velocity extracted from measured data for the PECVD film on 128° YX and YZ LiNbO₃ is shown versus normalized film thickness in Fig. 9. For both substrate cuts, the SAW velocity is faster under a silicon nitride film than on a free surface. As film thickness increases, the rate of increase of velocity with increasing film thickness decreases, implying that the film becomes less dense as it grows [3]. This decrease in film density with increasing film thickness was not observed in the measured film density values, perhaps due to measurement uncertainty. The difference in velocity dispersion observed for the two cuts may correspond to the difference in the compressional and shear elastic constants, c_{11} and c_{44} . The compressional elastic constant c_{11} is directly related to the SAW velocity, and is the factor most influenced by the application of a stiffening film such as silicon nitride. Thus the larger velocity dispersion observed for 128° YX LiNbO₃ may be due to the stiffening effect on the larger c_{11} of this cut.

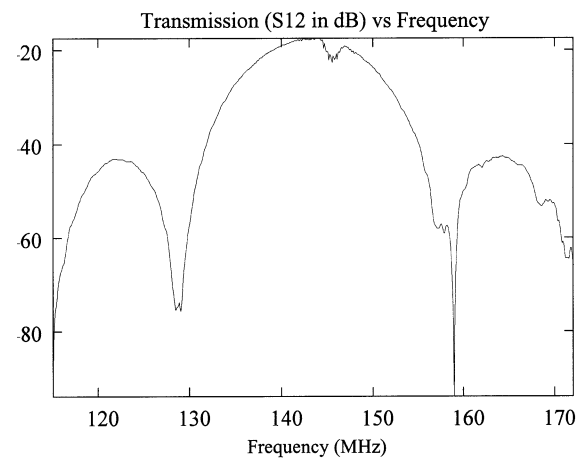


Fig. 10. Transmission data for a 100 strip silicon nitride reflector between two unweighted transducers.

Simple delay line devices at approximately 100 MHz center frequency were fabricated on YZ LiNbO₃ with a silicon nitride reflector between the transducers. The reflector was composed of 100 $\lambda/4$ silicon nitride strips. The transmission between transducers is shown in Fig. 10. The notch does not occur at the transducer center frequency due to the higher velocity of the SAW under the silicon nitride surface as compared to the SAW transducer velocity. Assuming a linear variation with respect to normalized film thickness (h/λ), the reflectivity per strip is calculated using linear regression as $0.7411(h/\lambda) + 0.0023$ for film thickness' between $0.006(h/\lambda)$ and $0.016(h/\lambda)$ [21]. No explanation has been obtained for the constant offset in this value of reflectivity. An approximate fit to the observed data which agrees with conventional theory for nonconductive steps is given by a reflectivity per strip equal to $0.95(h/\lambda)$. This reflectivity is slightly higher than grooves on YZ LiNbO₃, which have a reflectivity of approximately $0.67(h/\lambda)$ [22]. Connected and unconnected thin aluminum lines on YZ LiNbO₃ have a reflectivity of approximately 0.018 and 0.011, respectively [22]. These values are essentially independent of metal thickness, since piezoelectric shorting and electrical regeneration effects (for the unconnected lines) are the dominant reflection mechanisms, rather than mass loading or geometric discontinuities. It can be seen from the expressions above that reflectivities equal to or larger than those for metallic strips can be achieved on LiNbO₃ using moderate thickness' of PECVD silicon nitride films. Thus, such films can be used to provide reflective structures useful in a wide range of acoustic devices.

V. CONCLUSION

Silicon nitride films were deposited by a PECVD process using silane-ammonia as the reactant gas mixture. It is found that both the deposition and etch rates of the films are good indicators of film density and quality. By choosing the proper combination of deposition parameters, it is possible to achieve the desired deposition and etch rate characteristics for films for

SAW applications. These films have low acoustic attenuation and relatively high reflectivity, properties useful for acoustic devices. The process parameters used for depositing high quality silicon nitride films for SAW applications were presented. This study has shown good correlation with previously published work and indicates that high quality PECVD silicon nitride films should be able to be successfully grown on many different types of SAW substrates. Silicon nitride films could be used for passivation, frequency tuning, dielectric reflectors, waveguides, and other applications requiring a low loss dielectric film.

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Jacqueline H. Hines (S'88-M'88) earned her B.S. degree in applied and engineering physics from the Cornell University College of Engineering, Ithaca NY in 1984, and her M.S. degree in electrical engineering from the University of Central Florida, Orlando in 1988. Ms. Hines is currently conducting research for her Ph.D. dissertation at the University of Central Florida, Orlando.

As an undergraduate, Ms. Hines was involved in several research efforts, including work as a Research Assistant at Bell Labs investigating the feasibility of using polyacrylamide gels in the fabrication of optical fibers and work in the Theoretical and Applied Mechanics Division of Cornell University's College of Engineering on creep crack propagation modeling. From 1984 to 1988, Mrs. Hines served on active duty in the U.S. Navy, leaving active service at the rank of Lieutenant. Her primary responsibility was technical instruction in the areas of physics and reactor theory at Naval Nuclear Power School in Orlando, FL. Ms. Hines joined Sawtek Inc., Orlando, FL, in 1988, as a Research and Development Engineer. Her research activities have included the theoretical and software implementation of a time domain approach to analyzing diffraction effects in SAW devices, investigations into the effects of ionizing radiation on SAW devices, and work on high frequency devices. Mrs. Hines is currently the Technical Coordinator for Research and Development at Sawtek Inc.

Ms. Hines is the Junior Past Chairman and Awards Chairman for the IEEE Orlando Section. She has served as the IEEE Orlando Section Secretary, Treasurer, Vice-Chairman, and Chairman, and has filled several positions including Chairman in the Orlando Chapter of IEEE Ultrasonics, Ferroelectrics, and Frequency Control Society (UFFCS). She has served on the organizing committee for several conferences, including the 1991 Ultrasonics, Ferroelectrics, and Frequency Control Society Symposium and the 1995 NTC Conference, both held in Orlando, FL. Ms. Hines is a member of IEEE, Tau Beta Pi, and Phi Kappa Phi.



Donald C. Malocha (S'69–M'74–S'76–M'77–SM'84–F'93) earned his B.S. degree in electrical engineering and computer science and his M.S. and Ph.D. degrees in electrical engineering from the University of Illinois, Urbana, in 1972, 1974, and 1977, respectively.

From 1977 to 1978, Dr. Malocha was a Research Associate in the Coordinated Sciences Laboratory at the University of Illinois working on SAW technology. From 1978 to 1980 he was a member of the Corporate Research Laboratories at Texas Instruments, Dallas, working on SAW based modulator and convolver systems. From 1980 to 1982 he was the Manager of Advanced Product Development for Sawtek, Orlando, developing low loss transducer technology, data acquisition techniques, and bidirectional filters. In 1989 he was a Visiting Scholar at the Swiss Federal Institute of Technology and in 1990 he was a Visiting Member of the Technical Staff at Motorola's Advanced Components Technology Group, Phoenix, working on SAW and ACT technology. Presently, he is the Martin/St. Laurent Professor in the Electrical and Computer Engineering Department and group leader of the Solid State Devices and Systems Laboratory at the University of Central Florida, Orlando. His research group is currently working on surface acoustic wave (SAW) technology, CAD techniques, SAW based systems, and acoustic charge transport (ACT) devices.

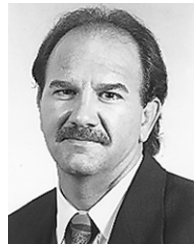
Dr. Malocha is an Associate Editor of the IEEE TRANSACTIONS ON ULTRASONICS, FERROELECTRICS, AND FREQUENCY CONTROL, vice-president for the UFFC society, and formerly a UFFC society elected Administrative Committee Member and Membership Chair. He serves on the Technical Program Committees of the Ultrasonic Symposium and Frequency Control Symposium, and was the General Chair of the 1991 Ultrasonics Symposium. He is past chair of the IEEE Orlando Section and the Orlando UFFC chapter. He has received the IEEE Orlando Section's Engineer of the Year, Educator of the Year and Outstanding Service Award in 1984, 1988, and 1989, respectively. He was awarded the UCF College of Engineering Excellence in Research award in 1986 and 1991, and received the Florida Governor's Award for Outstanding Contributions to Science and Technology in 1989. He is a member of the American Vacuum Society, Eta Kappa Nu, and Tau Beta Pi. He is a professional engineer, State of Florida, and holds six patents and several copyrights on computer software.



Kalpathy B. Sundaram ((S'76–M'79–S'79–M'80–M'87–SM'91) received the B.Sc. (Special) degree in physics from Kerala University, India, in 1970, the B.E. degree in electrical communication engineering from the Indian Institute of Science, Bangalore, India in 1973, and the M.Tech. and Ph.D. degrees in electrical engineering from Indian Institute of Technology, Bombay, India, in 1975 and 1980, respectively.

From 1980 to 1981 he worked as a Project Engineer at the Radar Project Center, Indian Institute of Technology, Bombay, India, and engaged in the development of X-Band phase shifters. In 1981 he joined the McMaster University, Hamilton, Canada, as a Post Doctoral Research Fellow and worked on Ge-GaAs heterojunctions, zinc oxide films for uv detectors, and CuInSe₂, CuInS₂ ternary materials for IR detectors till 1984. He joined the Opto-Electronics Inc., Oakville, Canada, in 1984 as a Research Scientist and worked on the development of lead chalcogenide materials for tunable diode lasers, diffused optical glass waveguides, and liquid phase epitaxial growth of InGaAs on InP for IR detectors till 1987. From 1987 to 1988 he worked as a Visiting Assistant Professor and from 1988 to 1993 as an Assistant Professor at the University of Central Florida, Orlando, FL. Since 1993 he has been an Associate Professor at the Department of Electrical and Computer Engineering, University of Central Florida, Orlando, FL. Presently his research interests include thin film materials and devices, optoelectronic materials, semiconductor device processing, porous silicon and micromachining.

Dr. Sundaram is a Senior Member of IEEE, Member of the American Vacuum Society and Electrochemical Society, Eta Kappa Nu, and Tau Beta Pi.



Kevin J. Casey received the B.S.E.T degree in 1989 from the University of Central Florida, Orlando, Florida.

In 1979 after being honorably discharged from the United States Air Force, he began working in the semiconductor industry for Texas Instruments, Houston, TX. He then moved to Harris Semiconductor in Melbourne, FL and then Martin Marietta Aerospace, Orlando, FL. Currently at the University of Central Florida, he is manager of the Solid State Devices and Systems Laboratory, where he is involved with process development of Surface Acoustic Wave (SAW) devices. His research interests include the development of a "narrow gap" process for (SAW) interdigital transducers (IDT), the fabrication of micro-cooling channels and heat-pipes in silicon, and the investigation of techniques used to measure the induced electric fields in an IDT using the electro-optic effects in SAW substrates. His current projects include the use plasma enhanced chemical vapor deposition (PECVD) to deposit silicon nitride films on single and multilevel SAW devices. He has coauthored papers on the characteristics of narrow gap transducers, imaging of electrostatic electric fields of SAW structures using electro-optic effects, diamond nucleation on graphite with a pure hydrogen feed, and deposition parameter studies and saw characterization of PECVD silicon nitride films on lithium niobate.

Kwang R. Lee, photograph and biography not available at time of publication.