IMPLANTED PLZT CERAMICS: A NEW HIGH-SENSITIVITY
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Summary

In recent papers¹⁻³ we presented results of our studies of photoferroelectric (PFE) image storage⁴⁻⁶ in H- and He-ion implanted PLZT (lead lanthanum zirconate titanate) ceramics which demonstrate that the photosensitivity of PLZT can be significantly increased by ion implantation in the ceramic surface to be exposed to image light. More recently, implantations of Ar and Ar + Ne into the PLZT surface have produced much greater photosensitivity enhancement. For example, the photosensitivity after implantation with 1.5×10^{14} 350 keV Ar/cm² + 1×10^{15} 500 keV Ne/cm² is increased by about four orders of magnitude over that of unimplanted PLZT. Measurements indicate that the photosensitivity enhancement in ion-implanted PLZT is controlled by implantation-produced disorder which results in marked decreases in dielectric constant and dark conductivity and changes in photoconductivity of the implanted layer.^{7,8} In this paper the effects of Ar- and Ar + Ne-implantation are presented along with a phenomenological model⁷ which describes the enhancement in photosensitivity obtained by ion implantation. This model takes into account both light- and implantation-induced changes in conductivity and gives quantitative agreement with the measured changes in the coercive voltage V_c as a function of near-UV light intensity for both unimplanted and implanted PLZT. The model, used in conjunction with calculations⁹ of the profiles of implantation-produced disorder, has provided the information needed for co-implanting ions of different masses, e.g., Ar and Ne, to improve photosensitivity.

Introduction

We first demonstrated in 1976 that optical images could be stored in transparent PLZT ceramics by exposure to near-UV light with photon energies equal to or greater than the band gap energy of ~ 3.35 eV.⁴ The image storage process relies on optically induced changes in the switching properties of ferroelectric domains, i.e., the intrinsic PFE effect.^{5,6} Stored images are nonvolatile but can be erased by uniform near-UV illumination and simultaneous application of an electric field. Another attractive feature of PLZT as an image storage medium is that the images can be switched from a positive to a negative or vice versa by switching the ferroelectric polarization from one saturation remanence to the other.^{10,11} Partial switching can be used to enhance contrast in regions of the stored image which were initially exposed at the same level by a technique called baseline subtraction.^{10,11}

Although high quality images, with contrast ranges of $\geq 100:1$ and spatial resolution of ~ 40 line pairs/mm, can be stored in ferroelectric (FE)-phase PLZT using the intrinsic PFE effect, relatively high exposure energies (~ 100 mJ/cm²) are required to store these images. This large exposure energy severely limits the range of possible applications of PFE image storage in unmodified PLZT. Since spatial modulation of the electrical and dielectric properties of the PLZT which contribute to formation of the image is confined to the near-surface UV absorption region, it follows that modification of the material to enhance the photo-

sensitivity should also be confined to the region adjacent to the surface. This reasoning provided the basis for the initial experiments with ion implantation. Our most recent experiments using co-implants of Ar and Ne have resulted in image storage exposure energy thresholds of ~ 10 μ J/cm² compared to ~ 100 mJ/cm² for unimplanted PLZT.

PFE Device Fabrication

The PFE image storage devices are extremely simple and easy to fabricate. The device consists merely of a thin plate of polished PLZT 7/65/35, which denotes a solid solution with the composition $\text{Pb}_{0.93}\text{La}_{0.07}(\text{Zr}_{0.65}\text{Ti}_{0.35})_{0.983}\text{O}_3$, with transparent indium-tin oxide (ITO) electrodes sputter-deposited on the two major faces. The ITO electrodes have a thickness of ~ 0.12 μ m and a resistivity of ~ 10 $\Omega/\square$.

Ion implantation was performed using a 300 keV Accelerators Inc. positive ion accelerator, and ion energies greater than 250 keV were achieved by implantation of doubly charged ions. For most of the data presented in the present paper the ions were implanted before the ITO electrodes were deposited; however, we have recently found that more uniform implants, and therefore more uniform domain switching, can be obtained if a thin (~ 200 Å) ITO layer is deposited prior to implantation to prevent charge buildup during implantation. In this latter case, additional ITO is deposited after implantation to yield a total electrode thickness of 0.12 μ m. A schematic illustration of the ion-implanted device is shown in Fig. 1. Typical device

Figure 1. Schematic illustration of a PLZT optical image storage device showing the implanted depth x_3 and the illuminated depth x_2 .

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dimensions are $25 \times 25 \times 0.25 \text{ mm}^3$, although devices with much larger areas can be readily fabricated. Typical implant and absorption depths are $x_3 \leq 1 \text{ }\mu\text{m}$ and $x_2 \sim 10 \text{ }\mu\text{m}$, respectively, and the remaining thickness $x_1 \sim 240 \text{ }\mu\text{m}$ is unaffected by either the implantation or illumination.

Characteristics of The Ion-Implanted Region

We have found that the ion-implantation-induced photosensitivity enhancement results from the disorder produced by energy the implanted ions impart to atoms of the PLZT. The primary effects of the disorder are to decrease the dielectric constant and the dark conductivity of the implanted region relative to the unimplanted dielectric constant and conductivity and to alter the photoconductivity of the implanted region. The substantial reduction of the dielectric constant indicates that the implanted region is probably no longer ferroelectric. The decrease in dark conductivity accounts for the observed increase in voltage required to switch the ferroelectric polarization.

The ionic depth distribution and the depth distribution of energy deposited into atomic and electronic processes can be accurately calculated using codes developed by Brice.⁹ Examples of the results of these calculations for 200 keV H and 200 keV He are shown in Figs. 2 and 3, respectively. The curves in Fig. 2

Figure 2. Calculated energy (eV/Å) depositions and proton depth distributions for 200-keV protons in PLZT 7/65/35 (7 at.% La; 65/35 zirconate to titanate ratio).

Figure 3. Calculated energy (eV/Å) depositions and He depth distributions for 200 keV-He ions in PLZT 7/65/35.

yield a projected range of $1.03 \text{ }\mu\text{m}$ for 200 keV H and the maximum in the energy deposited into atomic processes occurs at $0.9 \text{ }\mu\text{m}$. The 200 keV H implants deposit $\leq 0.5\%$ of the ion energy into atomic processes. For the more massive He ions, the projected range is $0.77 \text{ }\mu\text{m}$ and the maximum of the energy deposited into atomic processes occurs at $\sim 0.7 \text{ }\mu\text{m}$. For a given implant energy, the range of heavy ions decreases with increasing mass, but the fraction of implant energy deposited into atomic processes increases with increasing mass. This behavior is further illustrated in Figs. 4 and 5

Figure 4. Calculated energy deposition into atomic processes (displacement) and Ne depth distributions for 500 keV-Ne ions in PLZT 7/65/35.

Figure 5. Calculated energy deposition into atomic processes (displacement) and Ar depth distributions for 500 keV Ar ions in PLZT 7/65/35.

for 500 keV Ne and 500 keV Ar implants, respectively. Since ion implantation produced photosensitivity enhancement also depends on the thickness of the implanted layer, as will be discussed below, we have used co-implants of Ar and Ne to extend the disordered region to a depth of $\sim 0.5 \text{ }\mu\text{m}$ as shown in Fig. 6.

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Figure 6. Calculated energy deposition into atomic processes (displacements) for co-implanted 500-keV Ne + 350 keV Ar in PLZT 7/65/35.

Switching Characteristics

A hysteresis loop for an unimplanted, unilluminated PLZT sample 0.25 mm thick is shown in Fig. 7. For this thickness, the threshold field for domain

Figure 7. Hysteresis loop for unimplanted, unilluminated PLZT 7/65/35.

switching, $E_{th} \sim 3.5 \times 10^5$ V/m, occurs at an applied threshold voltage V_{th} of $\cong 100$ V. The coercive field $E_{C,u} = 4.1 \times 10^5$ V/m, where the polarization is zero, occurs at a coercive voltage $V_{C,u} \cong 120$ V. The subscript u in these notations indicates that the sample is unimplanted. When a similar sample is implanted with 2×10^{16} 200 keV H/cm², the dark coercive voltage $V_{C,i}(0) \cong 160$ V as shown by curve 1 of Fig. 8. At

Figure 8. Hysteresis loops for H-implanted PLZT 7/65/35 at various spatially uniform near-UV light intensities ranging from 30 $\mu\text{W}/\text{cm}^2$ (curve 2) to ~ 100 mW/cm² (curve 14). Curve 1 is for $I = 0$.

this applied voltage, the electric field in the underlying unimplanted region of the sample is equal to E_C of the unimplanted PLZT. This implies that $V_{C,i}(0) - V_{C,u}(0) \cong 40$ V is impressed across the implanted layer as a result of the decreased conductivity associated with implantation-produced disorder. For the Ar + Ne co-implanted sample, Fig. 9, the dark coercive voltage

Figure 9. Hysteresis loops for Ar + Ne co-implanted PLZT 7/65/35 at various spatially uniform near-UV light intensities ranging from 1 $\mu\text{W}/\text{cm}^2$ (curve 1) to ~ 100 mW/cm² (curve 15). Curve 0 is for $I = 0$.

(curve 0) has been increased to $V_{C,i}(0) \cong 275$ V. The voltage drop across the implanted layer in this sample is $V_{C,i}(0) - V_{C,u}(0) \cong 155$ V. This difference illustrates the effect of the greatly increased disorder in the Ar + Ne-implanted sample relative to that of the H-implanted sample. The curves numbered 1 through 15 of Fig. 9 were taken with increasing intensity I of near-UV illumination ranging from 1 $\mu\text{W}/\text{cm}^2$ (curve 1) to 98 mW/cm² (curve 15). The decrease in V_C with I is used for quantitative measurement of the photosensitivity as discussed below.

To store an image in the sample of Fig. 9, a switching voltage of, e.g., 150 V would be applied to the ceramic in the absence of illumination. Since this voltage is less than the threshold voltage for domain switching, no FE domains would be reoriented. The image to be stored would then be exposed on the implanted surface using near-UV light. As can be seen from the data in Fig. 9, the simultaneous application of UV light and the switching voltage will reorient domains

under the exposed regions of the ceramic. Since the domain switching is proportional to I , an image which faithfully reproduces the gray scale will be stored in the ceramic plate.

Phenomenological Model of Photosensitivity Enhancement

In this section we present a simple phenomenological model⁷ to describe both the photoinduced reduction of V_c which governs the storage process and the ion-implantation enhancement of the photosensitivity. Since the photon energy is greater than the band gap energy, the light will generate charge carriers and increase the conductivities in regions (2) and (3) of Fig. 1. To simplify the analysis, we first consider the case for unimplanted PLZT for which $x_3 = 0$ and $d = x_1 + x_2$. At the instant the voltage V is applied ($t = 0$), the electric fields in regions (1) and (2) are governed by

$$D_1 = D_2, \text{ or } \epsilon_1 E_1 = \epsilon_2 E_2 \quad (1)$$

where D_1, D_2 are electric displacements and ϵ_1, ϵ_2 are relative dielectric permittivities. For $t \rightarrow \infty$, the continuity equation reduces to

$$j_1 = j_2, \text{ or } \sigma_1 E_1 = \sigma_2 E_2 \quad (2)$$

where j_1, j_2 are switching current densities associated with domain reorientations, and σ_1, σ_2 are conductivities. For the experiments under consideration we are interested in the long time (essentially dc) behavior governed by Eq. (2).

To estimate the effect of illumination on the conductivity σ_2 , we assume that the photoexcited carrier concentration is dominated by intrinsic photoconductivity. Under this assumption the light-induced carrier concentration N will obey

$$dN/dt = aI - bN^2 \quad (3)$$

where aI and bN^2 govern carrier excitation and recombination, respectively. For constant I , $dN/dt = 0$, so that N , and the attendant changes in σ , are proportional to $I^{1/2}$. For intrinsic photoconductivity we thus write for region (2)

$$\sigma_2(I) = \sigma_1 + A\sqrt{I(x)} \quad (4)$$

If the conductivity is dominated by one type of carrier, A is given by $A = (a/b) e\mu$, where e and μ are the electronic charge and carrier mobility, respectively. It should be noted that $\sigma_2(I)$ is an exponentially decreasing function of depth x from the surface with a fall-off governed by the absorption length α^{-1} of $\sim 25 \mu\text{m}$ for $\lambda = 365 \text{ nm}$.

The applied voltage V is dropped across the sample according to

$$V = \int_0^{x_2} E_2(x) dx + \int_{x_2}^d E_1 dx \quad (5)$$

which, under the above assumptions, can be solved to yield $E(x)$ throughout the sample, as

$$V_u(I) = E_1 d \left\{ 1 + \frac{2}{\alpha d} \ln \left[\frac{\sigma_1 + A\sqrt{I_0} \exp(-\alpha x_2/2)}{\sigma_1 + A\sqrt{I_0}} \right] \right\} \quad (6)$$

when the field E_1 is taken to be constant throughout region (1). The subscript u denotes unimplanted PLZT, and I_0 is the light intensity at the surface. We

could have written a general expression for $E(x)$ throughout the depth d and solved Eq. (5) to yield an equation identical to Eq. (6) with x_2 replaced by d .

However, the purpose of dividing d into the regions x_1 and x_2 was to permit the solution to be expanded for small x_2 , where x_2 is the depth for which $\sigma_2 \gg \sigma_1$.

Although this expansion required an additional fitting parameter, the resulting equations are valuable for optimizing the device performance. We thus expand Eq. (6) for small x_2 to yield

$$V_u(I) \cong \left[1 + \left(\frac{\sigma_1}{\sigma_1 + A\sqrt{I_0}} \right) \frac{x_2}{x_1} \right] E_1 x_1 \quad (7)$$

Two interesting features are evident from Eq. (7). Since I decreases rapidly with x , the depth x_2 over which there is an appreciable change in σ changes with I . Secondly, in the above approximation, light-induced changes in the conductivity in region (2) have a second-order effect on the voltage required to attain a given field in the dark region (1). Physically this dependence reflects the fact that the low-conductivity bulk region governs the current through the sample. Once the conductivity of region (2) appreciably exceeds that of region (1), further increases in the conductivity of region (2) have only a small effect on the total current. Assuming that Eq. (7) holds at the coercive field for the present experimental conditions and postulating that the depth x_2 is governed by Eq. (4), within the framework of the above approximations Eq. (7) becomes

$$V_{c,u}(I) = E_{c,u}(0) \left(1 - \frac{x_2}{d} \right) \left[1 + \left(\frac{x_2}{d-x_2} \right) \left(\frac{\sigma_1}{\sigma_1 + A\sqrt{I_0}} \right) \right] \quad (8)$$

$$= V_{c,u}(0) (1 - B\sqrt{I_0}) \left[1 + \frac{B\sqrt{I_0}}{1 + A\sqrt{I_0}/\sigma_1(0)} \right],$$

where the approximate form of Eq. (8) derives from setting $x_2/(d-x_2) = x_2/d \equiv B\sqrt{I}$ in the second-order term. The subscript c denotes that $V_u(I)$ is evaluated at the coercive field $E_{c,u}$, and $E_{c,u}(0)$ is taken to be a constant parameter of the material, i.e. we assume a critical field is required to switch the unilluminated ceramic. B is defined by $B = 1/d$ times a parameter analogous to A in Eq. (4). The physical

significance of B is that $B\sqrt{I}$ is the value of x_2/d for which $E_2 \ll E_1$.

We next consider the effect of ion implantation. Ion implantation changes ϵ and σ in a well-defined near-surface ($\leq 1 \mu\text{m}$) region denoted as (3) in Fig. 1 and can have a marked effect on V_c for both the illuminated and the unilluminated conditions. Straight-forward solution of equations analogous to Eqs. (2) and (5) for $I = 0$ yields

$$V_{c,i}(0) = \left(1 + \frac{\sigma_1}{\sigma_3(0)} \frac{x_3}{x_1}\right) E_1 x_1, \quad (9)$$

where the subscript i denotes implanted PLZT, and σ_3 is taken to be uniform over the depth x_3 . From Eq. (9) it is apparent that the increase in the coercive voltage $V_{c,i}$ at $I = 0$ produced by ion implantation results from a decrease in the dark conductivity $\sigma_3(0)$ of region (3). For example, for 5×10^{14} 400 keV Ar/cm², which has $x_3 \approx 0.5 \mu\text{m}$, we find that $\sigma_1/\sigma_3(0) \approx 340$.

For $I \neq 0$, if we approximate the sample by three regions, analogous to a two-region approximation for unimplanted PLZT, Eqs. (2), (4), and (5) can be extended and solved to yield $V_{c,i}(I)$ in terms of the dark coercive field of region (1), $E_{c,u}(0)$. Assuming that the photoconductivity is intrinsic in the implanted region also, one finds

$$V_{c,i}(I) = V_{c,u}(I) + V_{c,u}(0) (1 - B\sqrt{I_0}) \left(\frac{1}{1 + C\sqrt{I_0}/\sigma_3} \right) \frac{\sigma_1 x_3}{\sigma_3 x_1}, \quad (10)$$

where $V_{c,u}(I)$ is given by Eq. (8) and C is analogous to A in Eq. (4) but refers to implanted material.

Discussion

If the microscopic parameters such as a , b , μ , etc., were known for PLZT, the parameters A, B and C introduced in the preceding section could be evaluated to calculate $V_c(I)$ from first principles. Unfortunately, the microscopic parameters are not known, so we treat A, B and C in Eqs. 8 and 10 as adjustable parameters to determine if the intensity dependence of V_c is adequately described by the model. $V_c(0)$ is directly measured at $I = 0$ so that A and B in Eq. (8) can be readily evaluated from the data. The solid curve for the unimplanted sample in Fig. 10 was calculated for:

Figure 10. Comparison between the model calculations and measurements of $V_c(I)$ for unimplanted, H-, Ar-, and Ar + Ne-co-implanted PLZT 7/65/35.

$A/\sigma_1(0) = 2.0 (\text{mW}/\text{cm}^2)^{-1/2}$ and $B = 0.022 (\text{mW}/\text{cm}^2)^{-1/2}$.

As can be seen for Fig. 10, Eq. (8) gives a good fit to the measured $V_{c,u}(I)$ for unimplanted PLZT throughout the intensity region $1 \mu\text{W}/\text{cm}^2 \leq I \leq 100 \text{ mW}/\text{cm}^2$ investigated.

Two variables of Eq. (10) which depend directly on the conditions of ion implantation are x_3 and σ_3 . To maximize photosensitivity, Eq. (10) indicates that the ion damage depth x_3 should be increased and that the dark conductivity $\sigma_3(0)$ in the implantation region should be minimized. The damage depth x_3 is increased either by implanting ions with lower mass or by implantation at higher energies; $\sigma_3(0)$ is reduced by maximizing implantation damage with more massive ions. Both of these dependences have been verified experimentally. The photosensitivity increase obtained with 500 keV Ar compared to 400 keV Ar (Fig. 10) illustrates the x_3 dependence. The lower photosensitivity for 200 keV He,³ which has greater x_3 than does 500 keV Ar, reflects the damage dependence.

To describe the dependence of the coercive voltage on I for ion-implanted PLZT, we use the parameters obtained for unimplanted PLZT together with $\sigma_1 x_3 / \sigma_3 x_1$ evaluated from Eq. (9) at $I = 0$ and C of Eq. (10) evaluated at some point on the $V_{c,i}(I)$ curve. Calculated $V_c(I)$ dependences for H, Ar, and Ar + Ne implants are compared with the measured dependences in Fig. 10. It is apparent from the curves in Fig. 10 for ion-implanted PLZT that Eq. (10) gives a reasonable description throughout the five decades in I which were measured. The curves for the Ar implants were calculated using the above parameters and $C/\sigma_3(0) = 0.80 (\text{mW}/\text{cm}^2)^{-1/2}$.

Identical parameters were used for the 400 and 500 keV Ar implants, with the exception of x_3 which was adjusted to account for the calculated differences in range of the implants.⁹ The fact that both curves give accurate fits to the data lends further support for this model.

The ion-implantation induced enhancement in photo-sensitivity is summarized in Fig. 11 where the change in coercive voltage ΔV_c is plotted as a function of I

Figure 11. Plots of the light-induced change in coercive voltage, $\Delta V_c = V_c(I) - V_c(0)$, versus light intensity for $1 \mu W/cm^2$ $\leq I \leq 100 mW/cm^2$. The image storage threshold occurs for $V_c \approx -25V$ and the threshold exposure energies for unimplanted H-, He-, Ar-, and Ar + Ne-coimplanted PLZT 7/65/35 are noted on the graph.

for different implanted ions. Empirically we find that the image storage threshold for a one-second exposure occurs at $\Delta V_c \sim -25V$, independent of the implanted species. The image storage threshold may thus be read directly from the curves in Fig. 11, and has been decreased from $\sim 85 mJ/cm^2$ in unimplanted PLZT to $\sim 45 \mu J/cm^2$ for co-implants of 5×10^{14} 500 keV Ar + 1.5×10^{15} 400 keV Ne. Very recently, we have further reduced¹² the threshold energy to $\sim 10 \mu J/cm^2$ with co-implants of 500 keV Ne and 350 keV Ar which yields a more uniform damage distribution than the co-implants reported in Fig. 11. We should also note that implantation with chemically active ions can be used to shift the absorption spectrum so that visible light can be used for image storage. For example, preliminary measurements indicate that light from an incandescent source can be used to store high quality images in Al-implanted PLZT. The measured photo-sensitivity for a sample implanted with 1×10^{15} 500 keV Al/cm² is decreased by less than an order of magnitude at 436 nm compared to the photosensitivity at 365 nm.

Conclusions

The relatively low photosensitivity associated with PFE image storage in unmodified PLZT severely limited the range of possible applications of PFE

Table I. A Summary of Properties of Selected Image Storage Media.

PROPERTIES PROCESSES	THRESHOLD SENSITIVITY E (ERGS/CM ²)	SPEED - ASA $S = E^{-1}$ (ERGS/CM ²) ⁻¹	SPECTRAL RANGE FOR STORAGE (NM)	SPECTRAL RANGE FOR READOUT (NM)	CONTRAST RANGE (OD)	SELECTIVELY ERASABLE	ELECTRICAL CONTRAST ENHANCEMENT	IMAGE VOLATILITY	MAXIMUM RESOLUTION (LINES/MM)	MAXIMUM IMAGE AREA (CM ²)	STORAGE VOLTAGE (VOLTS)
SILVER HALIDE FILM ⁽¹⁾ (ASA-100)	10^{-2}	10^2	VIS.	VIS.	~ 3	NO	NO	NO	~ 200		
ELECTROPHOTOGRAPHY ⁽¹⁾	1	1	VIS.	VIS.	~ 2	NO	NO	NO	~ 100		
Bi ₁₂ SiO ₂₀ - PROM ⁽²⁾ IMAGE STORAGE	50 100 100	2×10^{-2} 10^{-2} 10^{-2}	436 405 492	633	~ 3	YES	YES	YES	~ 80	16	4000
PLZT CERAMICS AR + NE IMPLANTED	100	10^{-2}	365	VIS.	~ 2	YES	YES	NO	~ 40	175	400
Bi ₁₂ SiO ₂₀ - PROM PHOTOREFRACTIVE ⁽³⁾	3×10^3	3×10^{-4}	442	633		NO	NO	YES	~ 100	16	4000
PLZT CERAMICS UNIMPLANTED	10^6	10^{-6}	365	VIS.	~ 2	YES	YES	NO	~ 40	175	400
DIAZO ⁽¹⁾	10^6	10^{-6}	VIS.	VIS.	~ 1	NO	NO	NO			

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(2) S. G. LIPSON & P. NISENSEN, APPLIED OPTICS 13, PP. 2052-2060 (SEPT. 1974)

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devices for image and information storage and display. By implanting inert gas ions into the surface of FE-phase PLZT, the photosensitivity of the PFE image storage devices has been increased by about four orders of magnitude. The exposure energy at the image storage threshold is $\sim 10 \mu\text{J}/\text{cm}^2$ for Ar + Ne-co-implanted PLZT compared to $\sim 100 \text{ mJ}/\text{cm}^2$ for unimplanted material. With this remarkable increase in photosensitivity, FE-phase PLZT is now the most sensitive,

nonvolatile, selectively-erasable image storage medium known to exist. Several important properties of various image storage media are listed in Table I. From the data given in the table, it is apparent that ion-implanted PLZT has about the same photosensitivity as $\text{Bi}_{12}\text{SiO}_{20}$ PROM devices, but the PLZT has the advantages of nonvolatile storage and appreciably larger maximum image storage area. These factors may be quite important for several memory and display applications.

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