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Electro-Optic LC Methods for Non-Destructive Testing of Solid Surfaces

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NLC testing for solid surfaces based on use of the flexoelectric effect in a spatially nonuniform field is described. Requirements to LC material of optimal sensitivity and spatial resolution are given. The method is illustrated by various examples of non-destructive testing.

Keywords: electro-optic effect, non-destructive testing, sensitivity, resolution

INTRODUCTION

Nematic Liquid Crystals (NLC) are known to be used in testing of solid surfaces.^{1,2} The advantageous features of the method such as being express, non-destructive, simple, cheap, as well as its high sensitivity and resolution, contribute to ever growing NLC application in industry.

The NLC surface testing procedure was first applied to analyze Integrating Circuits (IC) or their component layers, in particular a silicon dioxide layer. Papers^{1–5} describe use of the Dynamic Scattering Mode (DSM) effect for simple, quick, non-destructive testing of dielectric surfaces, search for rupture in metal IC buses, MOS substrate-short-circuited capacitors etc.⁶ When voltage is applied to an LC cell, there occur turbulent flows in places of imperfection. The method's sensitivity grows with voltage. This allows to visualize defects as small as about 0.5 μm . Study of defects on a dielectric surface using scanning electronic microscope showed that the turbulent regions coincide with the surface nonuniformities such as tiny convexities, etching holes, scratches, impurities and so on.^{5,7} The most promising among the NLC testing methods of solid surfaces, however, is the reported earlier^{2,3} procedure based on measurement of the NLC birefringence in the

vicinity of the defect. The first physical model of the method including optimization of the LC material parameters to obtain maximal sensitivity and resolution, as well as first practical applications for quality control of IC dielectric layers were reported in References 8–12.

NLC TESTING PROCEDURE

The cell used for the NLC testing method consists of a plate to be tested (electrode), a homeotropic NLC layer of specified thickness (normally 20 μm) and a glass plate with a conducting transparent layer of indium oxide (Figure 1).

Observation is carried out using microscope in polarized light under crossed polaroids. In the vicinity of defects—if any—on the surface investigated, the local electric field is nonuniform. Thus near the defect the director deviates from its original orientation. Accordingly, this results in a change of the birefringence and the defect becomes visible on a dark background (Figure 1).

In accordance with our concept of electro-optic effects in a spatially nonuniform field for low dielectric anisotropy $\epsilon_a = \epsilon_{\parallel} - \epsilon_{\perp}$, the NLC director response is thresholdless and fully determined by flexoelectric torque.¹³ The estimates^{10–13} show that the flexoelectric contribution predominates in the NLC electro-optic response in the NLC testing scheme (Figure 1) provided

$$U < 4\pi \left| \frac{e_{11} + e_{33}}{\epsilon_a} \right| \quad (1)$$

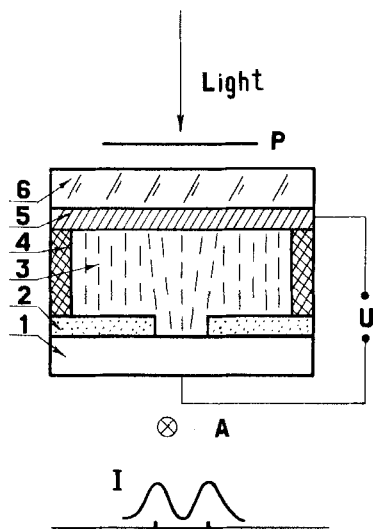


FIGURE 1 LC cell for NLC testing: 1, silicon plate with a dielectric film (2); 3, homeotropically oriented NLC; 4, dielectric (teflon) spacers; 5, glass plate with an electrode (6), P and A are polaroids, $I(U)$ is intensity visualized at voltage U .

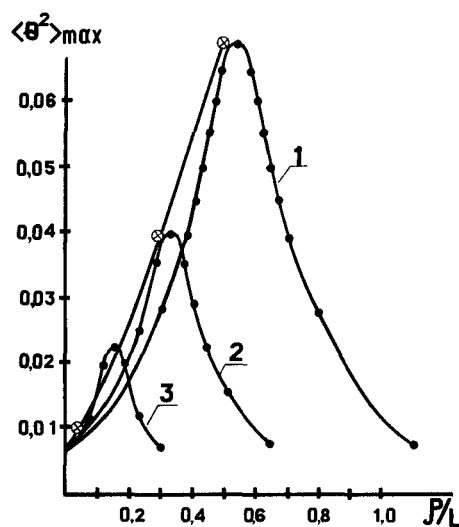


FIGURE 2 Measurements of director tilt near the defect $\langle \theta^2 \rangle_{\max}$ from distance ρ from the defect centre⁹ (L is radius of the defect): \otimes —theoretical estimations; 1 – $L = 20 \mu\text{m}$, 2 – $L = 10 \mu\text{m}$, 3 – $L = 4 \mu\text{m}$.

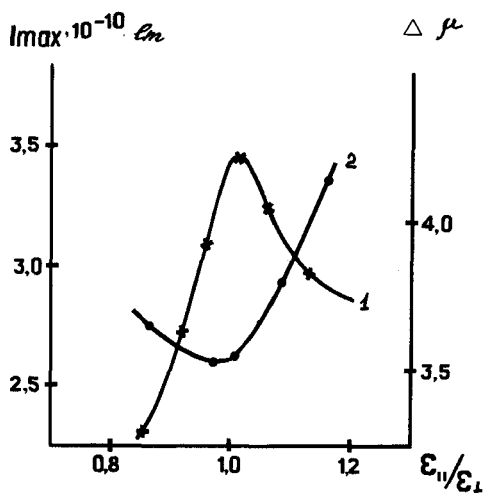


FIGURE 3 Experimental dependence of LC layer sensitivity (1) and resolution (2) on dielectric anisotropy.¹⁰

where U is value of the applied voltage, $e_{11} + e_{33}$ is the sum of the NLC flexoelectric coefficients.

Calculation of electro-optic characteristics for homeotropic NLC layer near the defect region (a cylindrical pore in a dielectric layer) proved that the sensitivity of the LC material is maximal when the so called “resonance condition” is valid^{9–12}:

$$\epsilon_{\parallel}/\epsilon_{\perp} = K_{33}/K_{11} = 1 \quad (2)$$

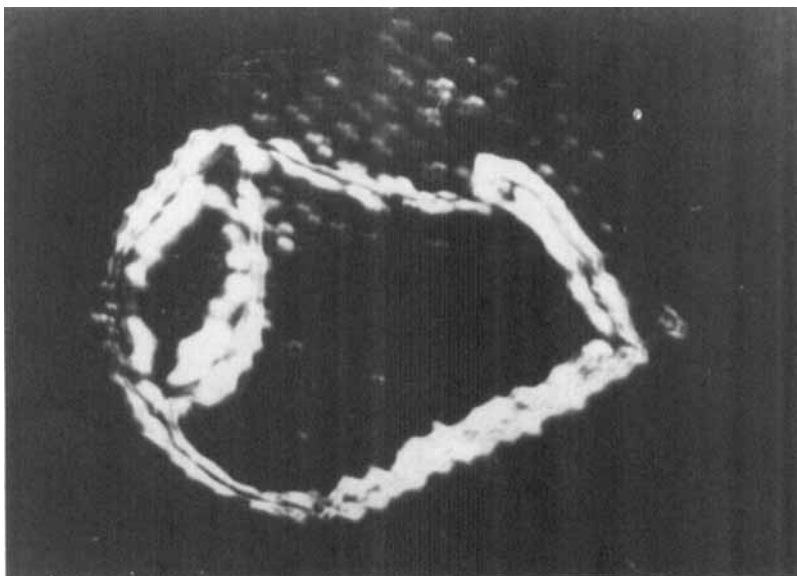


FIGURE 4 Pile-up of electrically active defects.

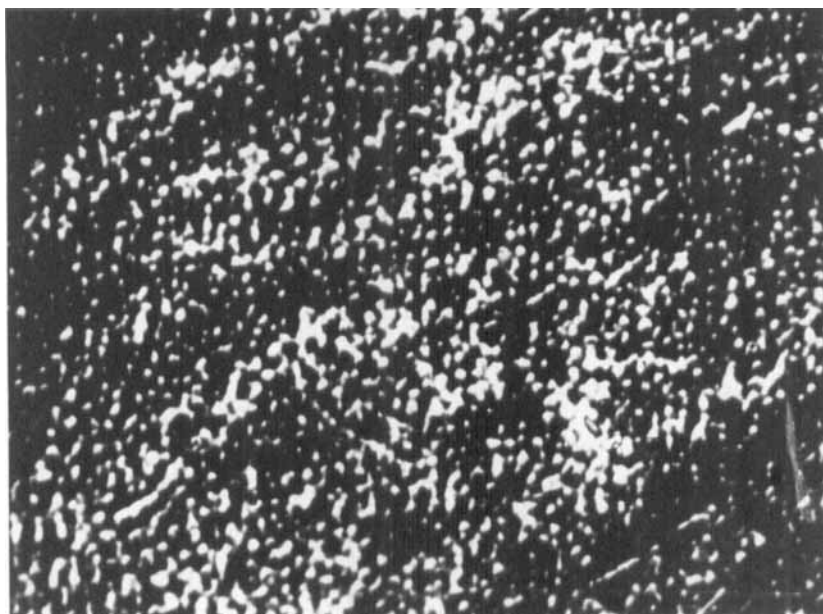


FIGURE 5 Si doped with islets of gold.

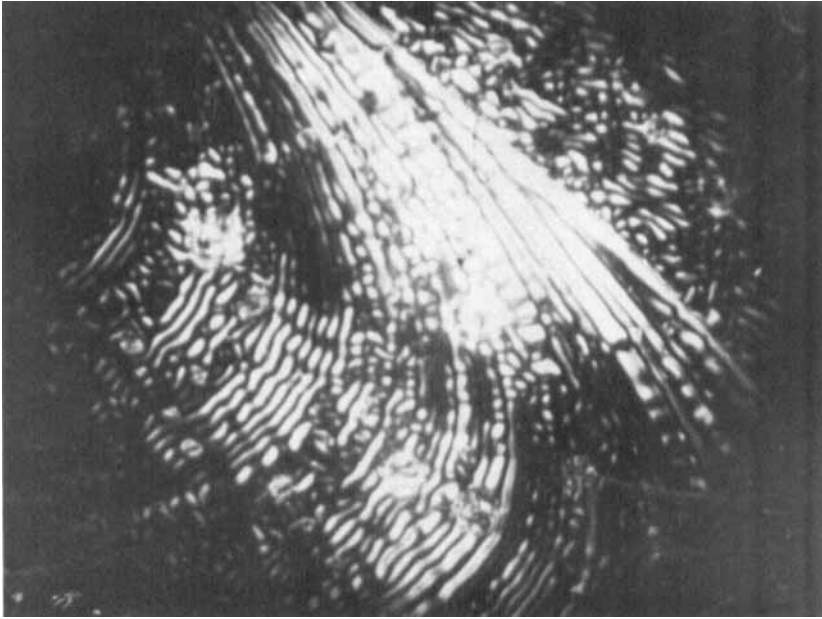


FIGURE 6 Fingerprint on a Si plate.

where K_{11} , K_{33} , ϵ_{\parallel} and ϵ_{\perp} are elastic and dielectric NLC constants. To check the theoretical conclusions, we conducted an experiment for NLC with different signs of dielectric anisotropy including $\epsilon_a \approx 0$. The magnitude of ϵ_a was varied near zero using different concentrations of dipole component with dielectric anisotropy $\epsilon_a < -1$.

Experimentally it was shown that the NLC optic response near the defect appears to be a double-humped curve (Figures 1 and 2) with maximal intensity near the edge of the defect where the nonuniformity of the electric field is maximal. Assuming that the magnitude of the peak intensity response per unit of applied voltage is proportional to sensitivity of the NLC testing and half-width of the response curve is its resolution, the optimum for the above characteristics (in agreement with the theory) is attained when condition (2) is satisfied (Figure 3). Thus according to our studies, the optimal NLC material in terms of sensitivity and resolution must meet the following requirements:

1. Maximal optical anisotropy $\Delta n = n_{\parallel} - n_{\perp}$;
2. Homeotropic alignment;
3. Observance of condition (2) at lowest possible elastic moduli K_{11} and K_{33} ;
4. Conductivity $\leq 10^9 \text{ Ohm}^{-1} \text{ cm}^{-1}$.

It is clear that conditions 1–3 enable maximal optic response of the NLC in the flexoelectric effect in a nonuniform field induced in the defect region, while condition 4 obtained experimentally guarantees absence of ions screening of the field.

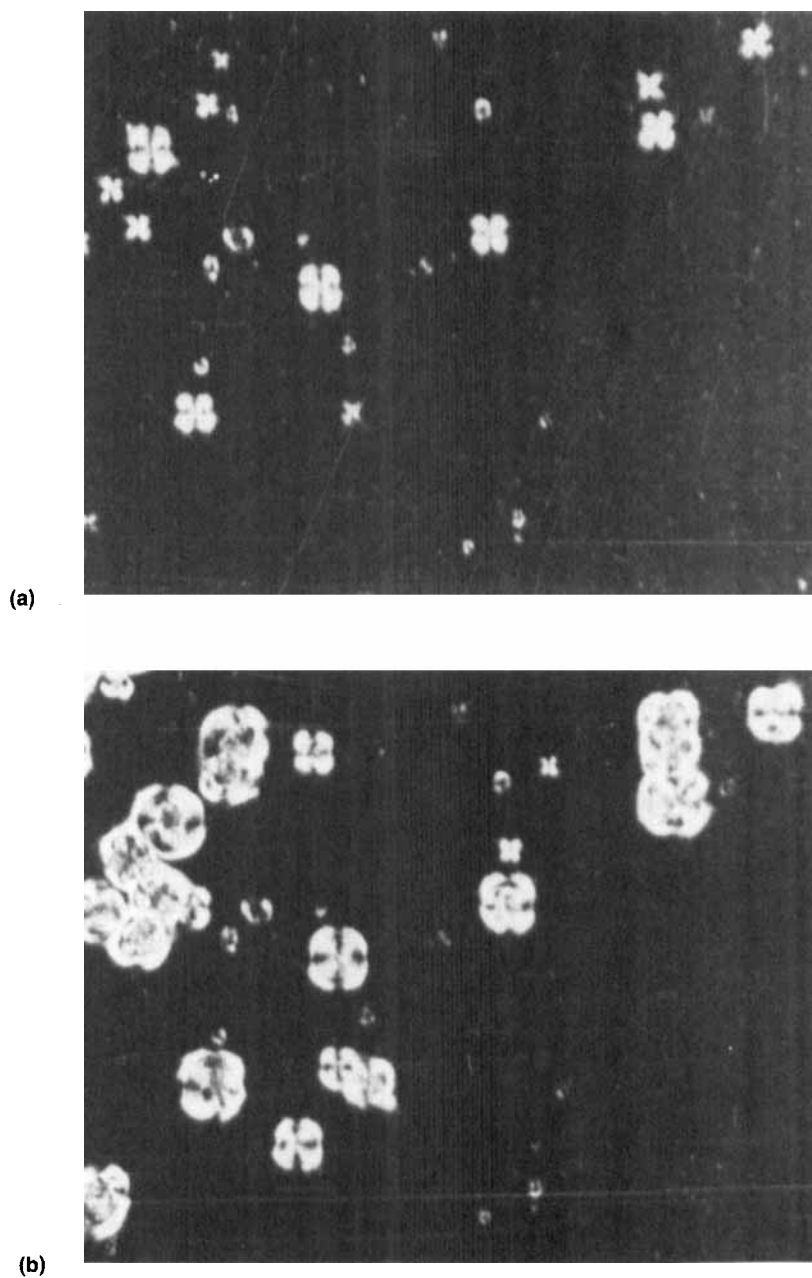


FIGURE 7 Visualization of defects in a dielectric layer: a) $U = 18$ V; b) $U = 22$ V.

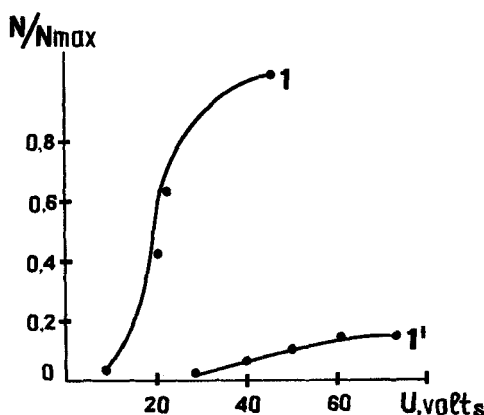


FIGURE 8 Relative quantity of detected defects versus voltage for various polarities¹⁰: 1, positive voltage (– at Si); 1', negative voltage (+ at Si).

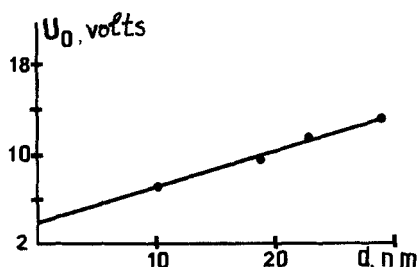


FIGURE 9 Threshold voltage of Fredericks transition in LC-dielectric-semiconductor structure versus layer thickness of dielectric Si_3N_4 .¹⁰

NLC TESTING APPLICATIONS

The NLC testing allows to check the quality of semiconductor plates (Si, InAs, InSb) in the following ways:

1. By detecting electrically active defects. Figure 4 shows electrically active dislocation pile-up on the surface of Si plates resulted from thermal treatment followed by sharp temperature drop. The density of the detected imperfections is $\approx 10^2 \div 10^3 \text{ cm}^{-2}$.
2. By visualizing rapidly diffusing dopants. Figure 5 shows a Si plate doped with islets of gold.
3. By detecting rough plate surface defects such as traces of cutting or grinding, scratches etc., as well as contaminants. Figure 6 shows a fingerprint on a Si plate surface.

NLC can be also used to locate defects of dielectric films applied in microelectronics (SiO_2 , Si_3N_4 , Al_2O_3 and so on). The NLC optic response in the vicinity of the defect resembles a Maltese cross (Figure 7a). Increase in voltage leads to growth of the cross size (Figure 7b) allowing to visualize more delicate defects.

The experiment shows that visualization of any defect on an oxide surface is

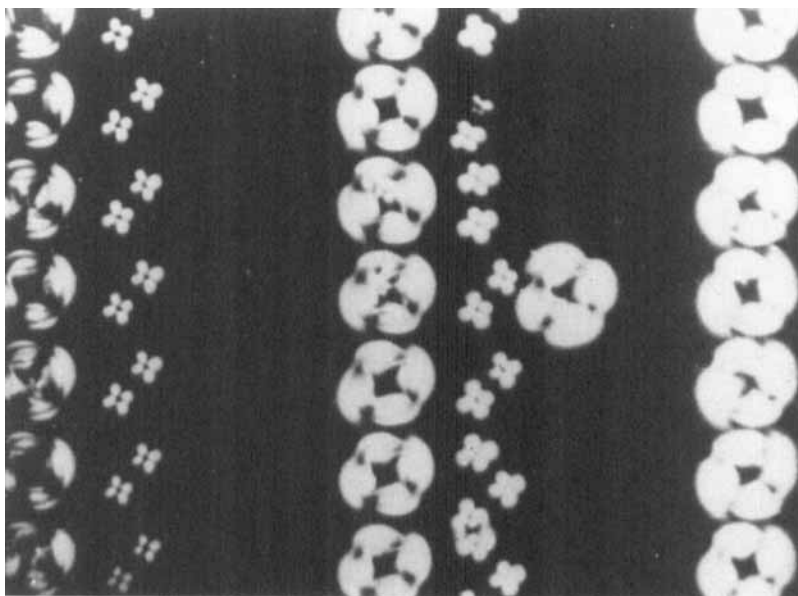


FIGURE 10 Measurement of oxidic residual layer thickness in IC contact windows.

dependent on polarity of the voltage applied to the LC cell (Figure 8). The amount of defects found at negative voltage (Curve 1'—a positive bias on Si) is significantly lower. Defects which can be visualized for both polarities of the voltage applied to the NLC are called bipolar, while those visualized only for positive voltage are called monopolar. Thus the NLC testing method can be also used to classify defects. The monopolar defects are recess pores, protrusions on silicon surfaces, SiO_2 film impurities and so on. The bipolar defects are open pores, protrusions on silicon surfaces penetrating through the dielectric layer, convexities on the oxide surface due to occurrence of cracks etc.

The NLC method can be applied to measure dielectric film thickness using the corresponding linear dependence of dielectric thickness on the magnitude of the threshold voltage for the Fredericks transition in the LC-dielectric-photoconductor structure (Figure 9).

Another successful application of the method is quality control at various stages of IC production, namely: to detect residual oxide in contact windows (Figure 10), short-circuited floating transistor gates, breaks in metallization of buses, leaks as well as to check operationability of devices assembled in casings (Figure 11).

Finally, the NLC method can be used for flaw detection of photoresist surface applied on silicon substrate or a SiO_2 layer at every treatment stage: coating followed by drying, development, redrying and so on.¹⁰

CONCLUSION

In conclusion let us point out some important advantages of the NLC procedure for quality control of various solid surfaces.

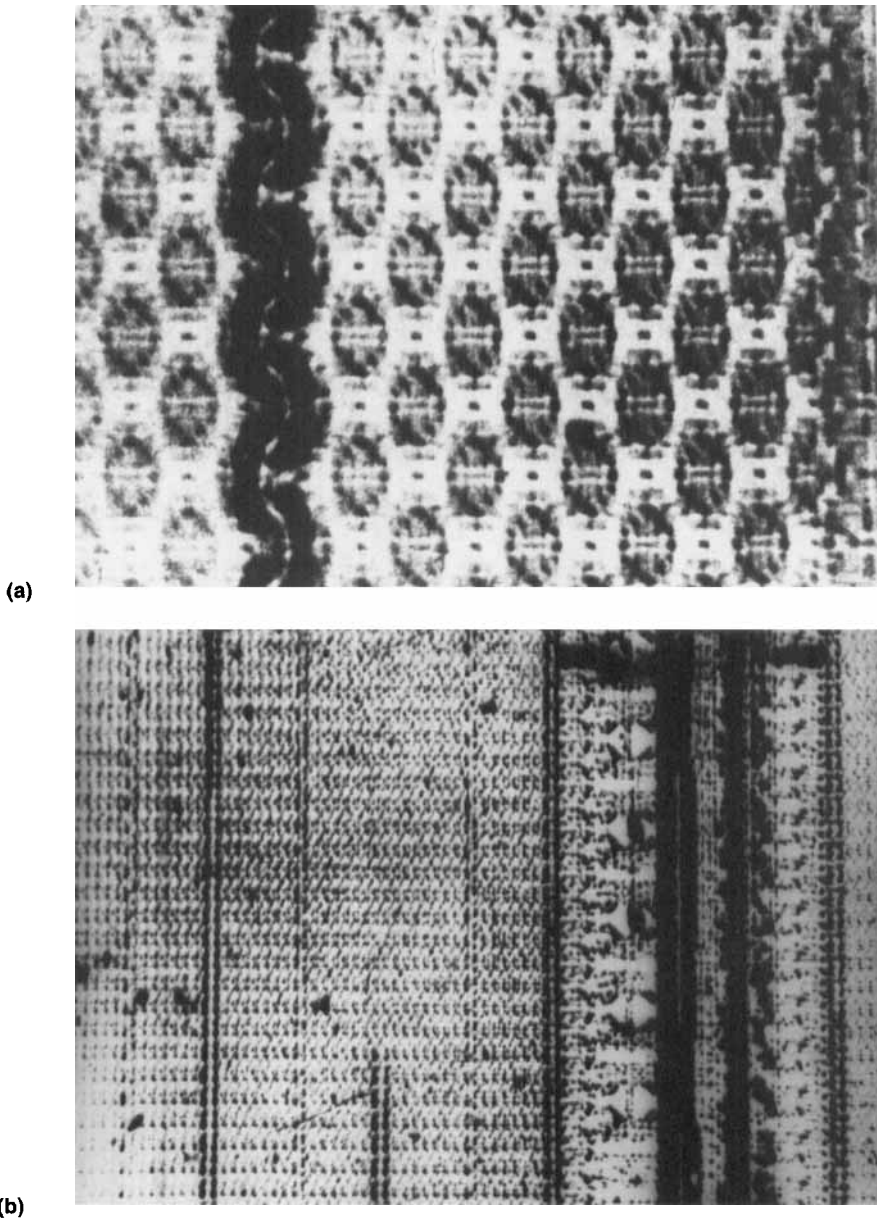


FIGURE 11 Detection of defects in IC elements: a) substrate-short-circuited chains of transistor gates, b) use of LC to control signals in finished MDS IC.

As compared to scanning electronic microscopy which is conventionally referred to as a non-destructive testing, the NLC method of flaw detection needs no sophisticated and/or costly equipment. At the same time it is simple and fast allowing to avoid irradiation by an electronic beam which might lead to charge accumulation and increase in leakage current in the devices being tested.

Comparing the NLC procedure with such known electrochemical method also needing no cumbersome or expensive facilities as electrographic, bubble and the so called copper-gelatine methods, we should mention the better resolution of the former. Thus, resolution of the traditional methods does not exceed a few microns for break-down voltages. In addition, their density of current passing through the defect (about 80 A/cm²) is several orders higher in magnitude than this for the NLC testing (about 10⁻⁷ A/cm²).

The maximal possible resolution of the NLC procedure obtained for pores in a SiO₂ layer about 100 nm thick was ≈0.01 μm at U ≈ 60 V. Such a high sensitivity of the method is due to the fact that the area of deformation in the LC layer considerably exceeds the defect in size. In other words, the LC layer acts as a magnifying lens.

Finally we note that visualization of defects in a NLC based on the reported here flexoelectric effect is significantly more efficient than in the DSM method because of the higher contrast, lower visualization voltages (3 to 4 times) and lower leakage current (about 10 times).

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