Supporting Information

Zirconia nanoparticles/ferroelectric liquid crystal composites for ionic impurities free memory applications

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Detailed experimental processes for the fabrication of liquid crystal sample cell

1. Photolithography

The photolithographic technique is used to transfer a desired pattern from a photo-mask to the substrate in liquid crystal (LC) sample cell fabrication. Photolithography involves several numbers of steps such as substrate cleaning, photoresist application, soft baking, mask alignment, UV exposure, pattern development. At first, the indium tin oxide (ITO) coated glass substrates were washed with soap solution to remove the traces of organic impurities and then cleaned with acetone to remove the dust particles. This process has been repeated thrice to ensure the proper cleaning of the substrates.

1.1 Photoresist application

After cleaning processes, the positive photoresist is coated over the surface of the substrate using spin coating unit. The spinning has been performed at 3000 rpm for 20 seconds. A uniform coating of photoresist of desired thickness is obtained by varying the spinning rate.

1.2 Soft baking

The process of preheating of the photoresist treated substrates before mask alignment is called soft baking. The excess solvents are removed from the photoresist coating during soft baking. The photoresist coatings become photosensitive only after the soft baking. The soft baking is done at 115 °C for 1 minute.

1.3 Mask alignment and UV exposure

After soft baking, the substrate is set against the photomask and exposed under the ultra violet (UV) light through the mask of desired pattern. The UV light was exposed for 4-5 minutes.

1.4 Pattern development

The UV exposed substrates were developed using developer of the positive photoresist and UV exposed part becomes soluble to the photoresist developer. The unexposed portion remains insoluble to the photoresist developer. Exposure of the UV light changes the chemical structure of the resist so that it becomes soluble in the developer. The UV exposed resist is then washed away by the developer solution, leaving windows of the bare underlying ITO material.
2. Etching

Etching is the process which is used to remove undesired ITO on the substrates after photolithography. We have performed wet etching. In wet etching, the ITO portions uncovered with photoresist get dissolved when immersed in a chemical solution. For ITO, the etchant can be \( HCl \) (15%) + Zn dust or \( HCl + HNO_3 \). Zinc dust is sprinkled uniformly into the dilute \( HCl \) (15%). After a few seconds, the masked glass substrates are immersed in the \( HCl \) (15%) + Zn dust solution. The chemical reactions involved during the etching process are as follows:

\[
Zn + 2HCl \rightarrow ZnCl_2 + 2H^+
\]

\[
SnCl_2 + 2H^+ \rightarrow Sn + 2HCl
\]

The nascent hydrogen produced during the reaction between the acid and zinc dust reduces the oxide coating on the glass substrate. Etching is stopped by dipping the glass substrates in water. After etching, the glass plates were cleaned with acetone to remove photoresist from unexposed part. Thereafter we checked the electrical continuity of developed pattern.

3. Alignment

We have given homogeneous alignment (in which the LC molecules are aligned parallel to the substrates) for the patterned ITO coated glass substrates. In order to get homogeneous alignment, we have used conventional rubbed polyimide technique. In this method, at first, a suitable polyimide (we used nylon 6/6) is coated uniformly on the substrate surfaces and then a smooth unidirectional rubbing is made after baking the polyimide coated substrates. The nylon 6/6 solution was prepared by mixing nylon 6/6 fibers, \( m \)-cresol, and methanol in the ratio 0.5:60:40. This solution was spin coated on the glass substrates at suitable rotational speed. The excess solvent is then evaporated by keeping the coated glass plates at 120 °C for 45 minutes. The polymer treated substrates were rubbed uni-directionally with a good quality velvet cloth. The rubbing direction was kept horizontal. We did anti-parallel alignment in LC sample cells.

4. Assembling, sealing and filling the cell

After performing the surface treatments for LC alignment, the ITO coated glass substrates were assembled in the form of a cell. During the assembling process, the Mylar spacers of thickness 6 \( \mu m \) were introduced between the substrates just outside the conducting portions to give proper cell
gap. The cell was then sealed using UV sealant at the four corners. UV sealant is applied all over the periphery of the cell and put under the UV source for 15 minutes. After proper sealing of the cell, connections were taken out from the two electrodes. The thickness of the cell was measured by capacitance measurement. In capacitance measurement, the air capacitance of the cell was measured from which thickness of the cell can be calculated using the formula

\[ d = \frac{\varepsilon_0 A}{C_0} \]  

(1)

where \( d \) is the thickness of the cell, \( \varepsilon_0 \) is the absolute permittivity (8.854 \( \times \) 10\(^{-12} \) C\(^2\) / N m\(^2\)), \( A \) is the conducting area (20.25 \( \times \) 10\(^{-6} \) m\(^2\)), \( C_0 \) is the air capacitance of the cell. After determining the cell gap, the pure FLC or ZrO\(_2\) doped FLC material were filled into the sample cell. For that at first, we set the oven temperature few degrees above the isotropic temperature of the FLC material used. A small quantity (~ 4 mg) of FLC material was placed onto the small opening between the plates and the cell was put in the oven. The LC material enters into the cell by means of capillary force after melting. The sample cell is then cooled slowly to room temperature.

**Thermal stability of zirconia nanoparticles/ferroelectric liquid crystal composites**

Figure shows the thermogravimetric analysis (TGA) results generated on pure and zirconia nanoparticles (ZNPs) doped ferroelectric liquid crystal (FLC). The plot shows the percent weight as a function of sample temperature for pure and ZNPs doped FLC in nitrogen environment. The TGA results show that the pure FLC material undergoes faster thermal degradation than ZNPs doped FLC. It can be mentioned here that ZNPs doped FLC sample is thermally stable than pure KCFLC 10S upto 225 °C.
**Figure:** TGA results obtained for pure and ZNPs doped FLC samples showing thermal stability.