



Investigation of Pyroelectric Effect generated by LiNbO₃ Crystals induced by integrated Microheaters

Shomnath Bhowmick

Tutor: Giovanni BreglioCo-Tutor: Giuseppe Coppola

XXIX Cycle- III year Presentation



General Information

- **1.** Name: Shomnath Bhowmick
- **2.** Bachelor Degree : *Electronics and Communication from JNTU*, *India* (2004-2009)
- **3.** Master's Degree : Nano electronics and Mechanics from Sheffield & Leeds, *UK* (2009-2011)
- **4.** Work Experience : *MEMS Engineer at i2n technologies private limited in Bangalore, India (2011-2012)*
- 5. IMM-CNR : Research fellowship on pyroelectric biosensor for pyrojetting (since 2012)
 - **University of Federico II, Naples :** PhD (ITEE) 29th cycle (since 2014)







Module and Seminar credits

Number Of courses	Credits Awarded
Euro Progettazione	3
National Instrument training	3
Three core issues for the internet: things, security and economic	2
GE2015 PhD school	4
AFM/EFM specialist course	4
An introduction to physics of nanostructures	4
Integrated Photonics	3
A lung-on chip to measure oxygen affinity of single red blood cells	4
Scientific Writing Course	5
Total	32

Number Of seminars	Credits	Date	Number of Hours (Hrs)
Luce e future	1.6	1/12/2014	4+4
Horizon 2020 I progetti Europei nella prospettiva di Un valutatore Eu (CNR)	0.8	11/11/2014	4
Biotechnologie industriali	0.8	30/01/2015	4
Secondo convegno nazionale sensori	1.6	19/03/2014	4+4
Engineering electromagnetic fields at the nanoscale	0.4	26/10/2014	2
Keys to getting your papers published	0.4	15/09/2015	2
Label-free imaging and characterization of bovine sperm cells: a combined holographic and spectroscopic approach.	0.8	18/06/205	4
Laser emission devices and methods for CARS-based spectroscopy of electroporated biological cells	0.8	22/05/2015	4
Development of an automated microfludic platform for multiplexing in flurosecence analysis	0.8	20/06/2016	4
Digitization, digital restoration, and visualization of antiquities: medieval manuscripts and scrolls from Herculaneum	0.4	04/03/2014	2
Setting Up a lattice Light-	0.4	01/06/2016	2
Frontier in single Molecule Manipulation and Imaging of DNA-protein transactions	0.8	22/01/2016	4
A lung-on chip to measure oxygen affinity of single red blood cells	0.4	11/06/2014	2
Quantum Simulation with Integrated photonics	0.8	17/03/2014	4
Applications of Optical Eigenmodes in biophotonics	0.8	25/03/2014	4
Porou silicon: From optoelectronics devices to an innovative bio-analytical tool	0.8	11/03/2014	4
Export of cargo from the Endoplasmic Reticulum is regulated by a control system	0.8	02/03/2016	4
Total	13.6		68

Other Activities

Conference/ workshops:

- Capri EOS tropical meeting 2015 17th September -19th September.
- Toriono fotonica conference 6th-8th may 2015.
- GE Siene Conference 22nd-24th June 2015
- Rome nano-italy conference 23/09/2015 (8hrs)
- Toulose France NMDC conference 09th-12th Octøber 2016

Abroad experience: South Korea (AFM/EFM)





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Tutor: Name Surname giovanni.breglio@u nina.i

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Tutorship:

AFM/ EFM for Maters students

Capri project: 4 hours (2 hrs theory+2 hrs practical) 08/09/2015 STEPFAR project : 4 hours (2 hrs theory+2 hrs practical) 21/09/2015 **CERVIA** project: 4 hours (2 hrs theory+2 hrs practical) 20/10/2015 STEPFAR project : 4 hours (2 hrs theory+2 hrs practical) 26/10/2015

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Motivation

- Pyroelectric effect ?
- Microheater Designs \rightarrow (LiNbO₃)
- Theoretical characterization (Thermal /Electrical)
- Fabrication Process
- Thermal analysis of Microheaters
- Electrical field analysis
- **PE analysis of LiNbO**₃ Metallic Probe point method
- Results and Discussions
- PE investigation Under Humidity Conditions
- Applications → (Nano -Pico droplet dispensing, Microneedles formation, Micro-spiral formation)
- Conclusion



Pyroelectric Effect



What is Pyro-electricity ?

- Pyroelectric effect is the ability of certain materials to generate a temporary voltage when they are heated or cooled.
 - This change in temperature induces a slightly movement of molecules within the material that changes their dipole moment.

Thus two oppositely charged faces are created and an electrical field across the material is established.

We investigate the pyroelectric effect in ambient conditions.





Pyroelectric effect applications

- Pyroelectric effect gives us the possibility to measure the power generated by radiation source such as:
 - Pyrometer
 - Infrared Imaging
 - Radiometry
- Other recent works on Pyroelectric effect are:
 - Lithography

"Patterned Pyroelectric Electron Emitters and their Feasibility Study for Lithography Applications", J. Appl. Vol. 42 pp. 3523-3525

Electron emission device

"Electron emission type infrared imaging sensor using ferroelectric thin plate", Sensors and actuators A: 97-98 (2002), 147-152

Nano particle alignment using pyroelectric effect

"Spontaneous Assembly of carbon-based chains in polymer matrix through surface charge templates", Langmuir 2013, 29, 15503-15510.

Nano droplet drawing

"Dispensing nano-pico droplets and liquid pattering by pyroelectrodynamic shooting", Nature technology, (2010) DOI: 10.1038/NNANO.2010.82

Microneedles

"Electro-drawn drug-loaded biodegradable polymer microneedles as a viable route to hydrodermic injection", Advc Funct. Matter. 2014, 24, 3515-3523



How to Generate Pyro-electricity ?



Microheater

Microscopic, Reliable, Cost effective, Shaping temperature distribution, Control temperature gradient, lab on chip, Array

Microheaters Design





Oven Pizza



Microheater Pizza

Microheaters Design

- Microheaters are small high power heaters with precise control, that can offer temperatures.
- A microheater produces heat by applying an electric current.
- Microheaters are used in Humidity sensor, thermo optical switches, chemical sensors, flow sensors etc.

In our work

- We realize four different microheaters structures on +Z surface of LiNbO₃ crystals.
- Advantageous: gives an advantage of confined temperature gradient, miniaturization of microheater can avoid heating the complete crystal, together with low power consumption for application-based sensors.



Theoretical Characterization Of µ-heater designs

COMSOL MULTIPHYSICS



Comso Multiphysic



COMSOL Multiphysics Simulation

Thermal Simulation

- The simulations were performed by coupling the power generation, due to the joule heating, with the heat conduction and dissipation into the device considering also the heat exchange with the surrounding air.
- The multiphysics simulation uses the electric current module in combination with the heat transfer module.
- The joule heating was calculated as a consequence of the input voltage, V_{pot} applied to the µH pads.
- In particular, the electrical conductivity of the μ H, σ was modelled using a temperature dependent equation:

Boundary conditions: a) fan shape microheater, b) terminal voltage, c) & d) Convective cooling boundary conditions on Microheater and LN surface, e) & f) Meshing configuration of Microheater and LN.

$$\sigma = 1/(\rho_0(1+a(T-T_0)))$$

 $\rho_{0} = resistivity$ a= temperature coefficient T₀= reference temperature

Parameter	Titanium	LiNbO ₃
Heat capacity at constant	129	628
pressure (C _p) [J/kg*K]		
Electrical Resistivity (ρ)	1 732	2.63×10^{14}
[μΩ m]	1.752	2.00^10
Density (rho) [kg/m³]	4506	4700
Thermal conductivity (k) [W/(m*K)]	16.8	5.6
Surface emissivity (ɛ)	0.35	0.60

Different material properties for LiNbO₃ and Titanium



COMSOL Multiphysics Simulation

Electric field Simulation

- The activation of pyroelectric effect by the microheater and the related induced electrical field was simulated using COMSOL™ Multiphysics.
- In particular, the steady state electric field formation between the -Z surface of LiNbO₃ crystal and a metallic probe tip was investigated using the equation:

 $V=d_{cr} * \gamma * \Delta T / \epsilon_{cr}$

 $d_{cr=}$ Crystal thickness (0.5mm) $\gamma = pyroelectric coefficient (8.2nC*cm^{-2*K^{-1}})$ $\epsilon_{cr} = dielectric constant (31)$



Design used for the electric field simulation between probe tip and –Z surface of LiNbO3 crystal.



Fabrication Process



IMM-CNR CLEAN ROOM

Microheater Fabrication Process

 The microheater designs were fabricated using lithography process on a LiNbO₃ crystal.





- Titanium --- E-beam
- Aluminum --- Sputtering
- ITO --- Sputtering

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Lift off process (Acetone bath)





Flow chart for fabrication



The schematic view of the fabrication process steps

	Track lenath	Microheater Resistance		
Design	(mm)	Titanium	Aluminum	ITO
		[Ω]	[Ω]	[k Ω]
Meander	17.6	373	21	71.3
Fan	31.97315	818	49.73	155.5
Spiral	26.644	707	42.74	133.2
S shape	11.89	137	11.42	25.98



Four different titanium micro-heater geometries: a) Meander, b) Fan, c) Spiral, and d) S-shape

Titanium was the winner among the other materials

Thermal analysis of Microheater



Thermal analysis of Microheater



COMSOL simulated thermal maps of the four different microheater designs: a) meander, b) fan, c) spiral and d) S-shape

Temperature transverse profile of the microheater designs along AA' at 350mW: a) meander, b) fan, c) spiral and d) S shape.



COMSOL

COMSC

MULTIPHYSICS

Thermal analysis of Microheater

FLIR SC7000 Series thermo camera.

- The temperature vs. power plot for the individual microheater estimated using COMSOL simulator and observed by Thermo-camera.
 - The Thermal map for individual microheater obtained using Thermo camera.









Meander shape

Fan shape Spiral shape

S-shape

COMSOL Multiphysics Simulation

Transient study

- In time dependent study, a rectangular-shaped voltage signal was applied to control the off-on-off switching of heater for different heating duration.
- It was observed in the experimental and simulated time period data that the slopes of heating and cooling were different.
- The rate of heating is stepper and the cooling is faster in experimental analysis compared to the simulation.
- Experimental and simulated transient analysis of Microheater designs with heater on-time of 4sec.



Experimental and simulated transient analysis of microheater designs COMSOL measurement with eater-on-time of a) 1sec, b) 50 sec.



Experimental and simulated transient analysis of microheater designs with eater-on-time of 4sec: a) thermo camera measurement, b) COMSOL measurement.

Theoretical analysis of microheater using COMSOL at different power dissipation with heater on time for 1 sec and 50 sec.

Among the four heater designs, fan shape heater produces the highest temperature due to its higher track length followed by spiral and meander, where S shape microheater produces a uniform heating zone on the -ZLiNbO₃ surface.

Electrical field analysis of Microheater with metallic probe



Electric field analysis



Electric field analysis along the XY plane (inset images) of each microheater designs: a) meander, b) fan, c) spiral and d) S shape.

Fan shape microheater yield to have a higher electrical field strength, this behaviour is attributed to the Temperature transverse profile distribution





Electrical field analysis for the fan microheater at different tip to -Z surface gap spacing: a) 0.1[mm], b) 0.5[mm], c) 0.75[mm] and d) 1[mm]



 ΔT is the temperature variation induced by the μ Hs driven with an electrical power of 300 mW

Electric field vs. probe tip to surface gap spacing for the microheater designs

Pyroelectric Emission (PE)



A metallic Point probe method

A metallic tip is bought near the -Z surface of LiNbO₃, while the other probe is used for grounding same surface.

When a temperature variation occurs due to the power dissipated by the microheater, a strong electric field is generated on the -Z surface of the LiNbO₃ due to the uncompensated charges.

The electric field created, resulting in the dielectric breakdown of air $(3 \times 10^6 \text{ V/m})$ between the metallic tip and crystal surface.

The dielectric breakdown of air makes it partially conducting and enabling the metallic tip to detect the emitted electron from the LiNbO₃ surface.



a) Schematic of experimental setup ,b) schematic of tip probe

Results and Discussions



PEE Analysis

Pyroelectric emission from of LiNbO₃ crystal is generally due to perturbation by the temperature variation of equilibrium state between spontaneous polarizations P_s inside the crystal and external screening charges (q_{sc}) on the crystal surfaces.

At equilibrium, all P_s are fully screened by q_{sc} and no electric field exists. Any excess or lack of screening charges (q_{sc}), relatively to P_s leads to the appearance of an electrostatic state from the uncompensated charges (q) that generates an electric field on the opposite surface of LiNbO₃, which is given by:

 $q = \Delta (P_{\rm s} - q_{\rm sc})$





At the equilibrium state qsc = Ps i.e. at room temperature ΔT

 $q = \Delta(Ps - qsc) = 0$

At
$$\Delta T > 0$$
, $q_{sc} > P_s$

 $q = \Delta(Ps - qsc) < 0$

Implying the uncompensated charges of q_{sc} is the source of electric field E on the Crystal surface.

• At $\Delta T < 0$, $q_{sc} < P_s$

 $q = \Delta(Ps - qsc) > 0$

Implying the uncompensated charges of P_s is the source of electric field E on the crystal surface.

Gap spacing analyzing between tip and -Z surface LiNbO₃

PEE from -Z surface of FAN shape

- The measurements were performed at different tip to surface gap spacing's $(100\mu m 3mm)$ during heating and cooling conditions at 10mHz.
- The maximum temperature observed at 10mHz (heater on time 50sec) was 80°C (from RT ~20°C) for an applied voltage of 12.24V.
- During cooling, significantly higher electron emission were observed from 100μm 1mm from microheater, while in the heating condition, causes low electron emission from 100μm-500μm and vanishes for a larger gap (over 1mm) distance.



the microheater designs



Dielectric breakdown

- To estimate the probability of electron emission from the -Z crystal surface, using a well known equation for the electric field Egap as a function of gap spacing between tip and surface is used:
- As the electric field generated due to the temperature variation slowly reaches the electric field strength of (3*10⁶V/m), the PEE decrease gradually and as its goes below the field strength of air, the PEE fades away.

Temporal distance between electrical peaks

- The current peaks were acquired by the dynamic redistribution of the electrical charges on the surface of $LiNbO_3$.
- It was also noted that the temporal distances between adjacent electrical peaks decreases during the initial phase of the thermal transient, while increases during complete thermalization of the sample.
- The dynamic current peaks was evaluated analyzing the temporal distance trend between electrical peaks during the whole thermal transient.



Exponential plot for the impulse occurrence in time during the: a) heating condition, b) cooling condition.



A fitted curve shows an increase in the distance between the peaks exponentially with time, which shows the dependency of the rate of heating and cooling on the PEE in a transient condition of the crystal.

Pyroelectric Investigation Under Humidity conditions





Current peaks obtained on oscilloscope using metallic probe at 0.1[mm] : a) Voltage signal applied to microheater to dissipate electrical power of 300mW, b) 54% Humidity, c) 50% Humidity, d) 47% Humidity, e) 45% Humidity, f) 44% Humidity and g) 42% Humidity.

Schematic of the humidity measurement setup

PE Investigation under Humidity conditions

47% Humidity

55% Humidity

65% Humidity

74% Humidity



Current peaks obtained on oscilloscope using metallic probe: a) 42%, b) 47%, c) 50%, d) 55%, e) 60%, f) 65%, g) 70% and h) 74% Humidity.

Humidity Increases so as the PE and vice versa

Applications



Microscale spiral formation by Pyroelectrospinning

We report on an innovative version of electro-spinning (ES) that we call micro-pyro-electrospinning (μ -PES) by controlling the electric field generated pyroelectrically by integrated microheaters (μ -Hs). The pyro-electrohydrodynamic process activated in this way can be used to manipulate soft matter and is able to generate true spiral patterns at microscale by a direct process.



(a) μ -PES set-up, (b) Optical Microscope image of a PMMA 9% micro-scale, (c) A slide view of the μ -PES process.



Fig: m-PES with visualization of the superposition of the m-PES spiral (grey) over a golden spiral produced geometrically. The distance h_{μ} is in the range of millimeters.







- Four test sample were used for Pyro-jetting application.
- Frequency : 25 [mHz]
- Microheater used : Fan shape
- Voltage applied: 9[V]
- Power dissipated: 300 [mW]
 - Sample used:
 - OIR 906
 - OIL
 - Water
 - PDMS









Microneedles Formation under pyroelectric effect

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- Microneedle Formation using fan shape microheater
- Frequency : 20 [mHz]
- Microheater used : Fan shape array (3× 1)
- Voltage applied: 9[V]

Sample used:

Power dissipated: 350 [mW]





Further work to produce array 10*10 microneedles Using Integrated µH



Conclusion

- The pyroelectric effect induced by integrated titanium microheaters was investigated
- Four different microheaters designs (Meander, Fan, Spiral and S shape) were fabricated on the +Z surface of a lithium Niobate crystal.
- Thermal and electrical analyses on the device were performed both theoretically and experimentally.
- The pyroelectirc effect was investigated using metallic probe setup.
- PE was also investigated under humidity conditions.

List of publications

List of publications

- S. Bhowmick, M. Iodice, M. A. Gioffrè, G. Breglio, A. Irace, M. Riccio, G. Romano, S. Grilli, P. Ferraro, L. Mecozzi, S. Coppola, O. Gennari, R. Rega, G. Coppola "Investigation of Pyroelectric fields generated by Lithium Niobate crystals through integrated microheaters" Journal of applied Physics,
- L. Mecozzi, O. Gennari, R. Rega, S. Grilli, S. Bhowmick, M. A. Gioffrè, G. Coppola, P. Ferraro "SPIRALS FORMATION AT MICROSCALE BY μ- PYROELECTROSPINNING" Soft Matter, May 2016, DOI: 10.1039/C6SM00156D
- O. Gennari, S. Grilli, S. Coppola, V. Pagliarulo, V. Vespini, G. Coppola, S. Bhowmick, M. A. Gioffrè, V. Ambrogi, P. Cerruti, C. Carfagna, P. Ferraro "SPONTANEOUS ASSEMBLY OF CARBON-BASED CHAINS IN POLYMER MARTIXES THROUGH SURFACE CHARGE TEMPLATES" Langmuir December 2013, 29, pp. 15503-15510, DOI: 10.1021/la403603d.

Conference Proceedings

- S Bhowmick, M Iodice, M Gioffrè, G Breglio, M Riccio, A Irace, G Romano, G Coppola: "Pyroelectric effect Investigation on LiNbO₃ crystal under humidity conditions using Microheater". 11th IEEE Nanotechnology Materials and Devices Conference (2016), Toulouse, France. DOI: 10.1109/NMDC.2016.7777087
- L. Mecozzi, O. Gennari, R. Rega, S. Grilli, S. Bhowmick, M. A. Gioffrè, G. Coppola, P. Ferraro "SPIRAL FORMATION AT MICROSCALE BY M-PYRO-ELECTROSPINNING" AIP Conference Proceedings 1736, 020079 (2016); Naples, Italy.
- S. Bhowmick, M. Iodice, M.A. Gioffrè, G. Breglio, M. Riccio, A. Irace, G. Romano, G. Coppola "PYRO-ELECTRO-THERMAL ANALYSIS OF LINBO3 USING MICROHEATERS" Fotonica AEIT Italian Conference on Photonics Technologies (2015), Turin, Italy.
- S. Bhowmick, O. Gennari, S. Grilli, V. Vespini, G. Gentile, V. Ambrogi, P. Cerruti, M. A. Gioffrè, C. Carfagna, P. Ferraro, S. Coppola, V. Pagliarulo, G. Coppola "CARBON BASED WIRES ASSEMBLING USING PYROELECTRIC EFFECT CONTROLLED BY MEANSOF TITANIUM M-HEATERS" 1st EOS Topical metting on Optical Microsystems (OµS' 13), Capri, Italy

References:

G. Velmati, N. Ramshanker and S. Mohan, "2D simulations and electro- Thermal Analysis of micro-Heater Designs Using COMSOL for Gas Sensor Applications", Proceedings of the COMSOL Conference 2010, India.

El Mostafa Bourim, Chang-Wook Moon, Seung- Woon Lee, Vadim Sidorkin, In Kyeong Yoo, "Pyroelectric electron emission from –Z face polar surface of lithium niobate monodomain single crystal", Journal of Electroceramics, Volume 17, Issue 2-4, pp 479-485, Dec. 2006, doi: 10.1007/s10832-006-0387-y.

Darhuber, Anton A., Troian, Sandra"Thermocapillary actuation of droplets on chemically patterned surfaces by programmable microheater arrays", Journal of Microelectromechanical Systems, vol.12, no.6, pp.873,879, Dec. 2003, doi: 10.1109/JMEMS.2003.820267.

Ferraro P., Coppola S., Grilli S., Paturzo M. and Vespini V., "Dispensing nano-pico droplets and liquid patterning by pyroelectrodynamic shooting", Nature Nanotechnology, 5, 429–435, (2010), doi: 10.1038/nnano.2010.82.

V. Vespini, S. Coppola, S. Grilli, M. Paturzo and P Ferraro, "Milking liquid nano-droplets by an IR laser: a new modality for the visualization of electric field", Meas. Sci. Technol., 24 045203, (2013), doi: 10.1088/0957-0233/24/4/045203.

Thank You

Questions?

