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Abstract Book

PRESENTATIONS

(Presentation) SHAPE-CONTROLLABLE SYNTHESIS OF HYBRID STRUCTURES BY 3D HYDRODYNAMIC FOCUSING METHOD

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Micro-/nano- structures (MNS) have shown potential applications in electronics, materials science and chemical, physical and biomedical fields.[1] In micro/nano scale, the size or shape change can affect the materials properties, such as optical, chemical, electrical properties.[2] Therefore, it is very important to fabricate MNS with uniform size and shape.

Conventional method, which simply mix reagents by vortex mixing, is difficult to control the size and shape distribution of the product. During reaction, the addition of reagents, rate of stirring, local temperature and concentration fluctuations can affect the forming process of the product. Conventional method cannot make good control of these parameters, ending up to broad size and shape distribution of products.[3]

In microfluidic systems, the mixing of reagent is caused by diffusion only, because of the laminar flow in the micro scale channels. Therefore, microfluidic hydrodynamic focusing (HF) system has been used to precisely control the convectivediffusive mixing of the reagents. In the absence of turbulence, co-flowing streams carrying the reagents form a very well defined interface. Reaction and assembly take place directly at the interface as well as in the diffusion cone of the reactants. Compared to the conventional "bulk mixing" methods, microfluidic device can achieve reproducible, well-controlled mechanical and chemical conditions, leading to superior quality of the formation of MNS.[4]

Two-dimensional (2D) HF provides horizontally uniform environment (flow rate, pressure, shear stress, *ect.*) in the reaction region, but variety parameters in the vertical dimension, which still limit the performance of 2D HF system in synthesis application. Additionally, in 2D HF system, the reaction region contacts the top and bottom walls of the microfluidic channels. The aggregation of the product on the channel wall results in clogging of the channels, which substantially reduces the robustness and stability during synthesis process, and finally causes the failure of the device.[5]

Three-dimensional (3D) HF method can provide uniform environment both horizontally and vertically and confined the reagent streams at the center of the channel. Multi-step photolithography and assembly protocols have been developed for fabricating the 3D HF devices. However, these methods require either tedious assembly of individual components or multiple alignments and exposures during mold fabrication. These limitations inevitably increase the cost and complexity of the device and ultimately hinder their applicability. Our group has developed a simple single-layer planar microfluidic device based on a novel fluid manipulation technique named "microfluidic drifting".[6] This method is effective, robust and does not require any extensive fabrication technique other than standard soft lithography.

In this paper, we synthesized TTF-Au hybrid structures with different shape and size by using the "micro drifting" based 3D HF devices. By tuning the flow conditions, different structures, such as wire, rod, particles, flower-shape, multi-layer and triangle can be achieved. The products fabricated by 3D HF showed narrower size distribution compared to the one fabricated by "bulk" mixing. It is also found that, in microfluidic channel, even though the ratio between reagents as constant, by changing the flow rate, different structures can be fabricated.

A POWER FLUX DENSITY AND SYNCHRONISM CO-ANALYSIS FOR CUMULATIVE HIGH HARMONIC GENERATION IN A PLATE

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Abstract:

In order to maintain the best possible structural integrity it is highly desirable to detect damage at the smallest scale possible. Doing so with periodic nondestructive inspection or continuous structural health monitoring enables tracking damage evolution over the service life of the structure, which can be used in conjunction with prognostics for condition based maintenance and improved logistics. Generally speaking, linear ultrasonics with bulk waves can detect anomalies of the order of the wavelength. Ultrasonic guided waves can do significantly better by properly selecting a mode with wave structure sensitive to the defect. Nonlinear ultrasonics, where the received signal containing the information of interest is at a different frequency than the emitted signal, can provide sensitivity to micro-structural changes. The generation of measurable higher harmonics is extremely useful because these harmonics are sensitive to the very micro-structural features that cause the nonlinear elasticity, which means that they can be related to not merely macro-scale defects but precursors to them, such as dislocation density, nucleated voids, and precipitate coarsening.

In this research, the nonlinear wave equation is decomposed into a fundamental wave field and a secondary wave field based on a perturbation method. The fundamental wave field is solved by the hybrid SAFE and GMM method. The secondary wave field is solved by the normal mode expansion (NME). Then a thorough analysis of power flux from the fundamental plate wave modes to the secondary wave fields has been conducted, which includes fundamental SH wave field to secondary SH wave field, fundamental SH wave field to secondary Lamb wave field, fundamental Lamb mode to secondary SH wave field, and fundamental Lamb wave field to secondary SH wave field. It is concluded from the analysis that only symmetric Lamb modes can be cumulative along the propagation distance, anti-symmetric and SH wave modes cannot be in resonance with any type of fundamental wave field. Both SH and Lamb mode fundamental fields have the potential to generate cumulative second wave fields. A comprehensive analysis of phase matching conditions has also been conducted, from which we conclude that the synchronism points can only occur with special

phase velocities (longitudinal wave speed C_L ,

shear wave speed C_T , Raleigh wave speed C_R

or Lamé wave speed $C_{Lame'}$), or the crossing

points of fundamental wave modes or secondary wave modes. Then a method for the optimum fundamental excitation has been proposed based on the co-analysis of power flux density and synchronism conditions. The synchronism points in high power flux density area will be the optimum fundamental excitation for a cumulative secondary wave field. Experiments using an SH mode (SH 1 at longitudinal wave speed) and a Lamb mode (S1 at longitudinal wave speed) fundamental excitation have been performed to verify the co-analysis method. A increase of qualitative linear nonlinear parameter has been observed for both SH wave and Lamb wave experiments. It is also observed that the nonlinear parameter of S1 fundamental excitation is larger than that of SH1

fundamental excitation at the same point. This can be explained by the higher power flux from S1 fundamental field than that of SH1 fundamental field to S2 secondary wave field. Combining a cumulative harmonic with the penetration power of guided waves could be beneficial for nondestructive evaluation and eventually structural health monitoring.





Figure 1. Internal resonance plot for (a) fundamental SH wave generation and (b)Lamb wave generation

[1] M. X. Deng. Cumulative second harmonic generation of Lamb mode propagation in a solid plate. Journal of Applied Physics 85(1999) 3051-3058.

[2] W. J. N. de Lima, M. F. Hamilton. Finite-amplitude waves in isotropic elastic plates. Journal of sound and vibration, 265(2003) 819-839.

[4] M. F. Muller, J. Y. Kim, J. M. Qu, L. J. Jacobs. Characteristics of second harmonic generation of Lamb waves in nonlinear elastic

[3] A. Srivastava, F. L. di Scalea, "On the existence of antisymmetric or symmetric Lamb waves at nonlinear higher harmonics," Journal of Sound and Vibration, 323(2009) 932-943.

plates. J. Acoust. Soc. Am. 127(4) (2010) 2141-2152.

ENHANCED ABSORPTION OF LIGHT DUE TO MULTIPLE SURFACE-PLASMON-POLARITON WAVES

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For the last three decades, research to bring down the cost of photovoltaic (PV) solar cells has gained a huge momentum and many techniques to increase the efficiency of light harvesting by solar cells have been investigated. Among other methods [1], the use of plasmonic structures to enhance the absorption of light by PV solar cells has been studied [2]. The basic idea is to to have periodic texturing of the metallic backing layer of a thin-film solar cell to help excite surface-plasmon-polariton (SPP) waves. As the partnering semiconductor in these studies has been homogeneous, only one SPP wave (of *p*-polarization state) at a given frequency can be excited [3, 4] leading to modest gains in the absorption of light.

Recently, it has been shown that multiple SPP waves of different polarization states, phase speeds, and attenuation rates can be guided by the interface of a metal and a dielectric material that is periodically nonhomogeneous in the direction normal to the metallic/dielectric interface [5]. Also, multiple SPP waves can be excited using a grating-coupled configuration. Therefore, I set out to investigate if light absorption can be enhanced due to the excitation of multiple SPP waves by a surface-relief grating on the metallic backing layer in a PV solar cell with a periodically nonhomogeneous semiconductor. For this purpose, a boundary-value problem of reflection by a surface-relief grating coated with a periodically nonhomogeneous semiconductor was formulated and solved numerically.

Let me consider the boundary-value problem shown schematically in Fig. 1. The regions z < 0 and $z > d_3$ are vacuous, the region $0 \leq z \leq d_1$ is occupied by the semiconductor partnering material with relative permittivity $\epsilon_d(z)$, and the region $d_2 \leq z \leq d_3$ by the metallic partnering material with spatially uniform relative permittivity ϵ_m . Moreover, 2Ω is the period of periodic nonhomogeneity of the semiconductor material. The region $d_1 < z < d_2$ contains a surface-relief grating of period L along the x axis with the relative permittivity $\epsilon_g(x, z) = \epsilon_g(x \pm L, z)$.



Figure 1: Schematic of the boundary-value problem solved using the RCWA.

The total absorptance A_p (for *p*-polarized incidence) and A_s (for *s*-polarized incidence) for this structure waas calculated using rigorous coupled-wave analysis. Plots of the absorptances A_p and A_s vs. θ are provided in Fig. 2 for $L_g = 0$ and $L_g > 0$. Two A_p -peaks, at $\theta \simeq 12^{\circ}$ and 25.1°, in Fig. 2(a) are independent of the thickness of the semiconductor layer. One of the relative wavenumbers of Floquet harmonics at these values of θ are close to the relative wavenumbers obtained by the solution of the canoni-



Figure 2: Absorptances (a) A_p and (b) A_s vs. the angle of incidence θ , when $\Omega = 200$ nm, $d_3 - d_2 = 30$ nm, and $\lambda_0 = 620$ nm. Also, (a) L = 170 nm, and (b) L = 200 nm. Each vertical arrow indicates the excitation of an SPP wave.

cal boundary-value problem, so these peaks represent the excitation of *p*-polarized SPP waves. The spatial profiles of the *x*-component of the time-averaged Poynting vector for these two *p*-polarized SPP waves, provided in Fig. 3(a), support the conclusion that *p*polarized SPP waves are excited. Let me note that the *p*-polarized SPP wave that is excited as a Floquet harmonic of order n = 1 propagates in the $+\hat{\mathbf{u}}_x$ direction while the SPP wave that is excited as a Floquet harmonic of order n = -1 propagates in the $-\hat{\mathbf{u}}_x$ propagation.

The excitation of two *p*-polarized SPP waves is accompanied by a very significant increase in the absorptance for a *p*-polarized incident plane wave, as is evident from the comparison of the absorptances for $L_g = 0$ and $L_g > 0$ in Fig. 2(a). Similarly, the excitation of an *s*-polarized SPP wave in the gratingcoupled configuration is correlated with a significant increase in the absorptance in Fig. 2(b).

Let me note that the excitation of waveguide modes also plays a role in the enhancement of absorption in the grating-coupled configuration. However, geometric parameters of the surface-relief grating and the periodically nonhomogeneous semiconductor layer shall have to be optimized carefully in order to obtain an overall enhanced absorption of the insolation flux over the 400–1200-nm wavelength range.



Figure 3: Variation of the x-component of the timeaveraged Poynting vector P_x along the z axis at x = 0.75L for (a) two p-polarized incident plane waves and (b) an s-polarized incident plane wave, at the θ -values of the absorptance peaks identified in Fig. 2 by vertical arrows.

References

- A. Gombert and A. Luque, "Photonics in photovoltaic systems," Phys. Stat. Sol. A, 205, 2757– 2765 (2008).
- [2] V. E. Ferry, L. A. Sweatlock, D. Pacifici, and H. A. Atwater, "Plasmonic nanostructure design for efficient light coupling into solar cells," Nano Lett., 8, 4391–4397 (2008).
- [3] S. A. Maier, *Plasmonics:* Fundamentals and Applications, Springer, New York, USA (2007).
- [4] J. A. Polo Jr., and A. Lakhtakia, "Surface electromagnetic waves: A review," Laser Photon. Rev., 5, 234–246 (2011).
- [5] M. Faryad, and A. Lakhtakia, "On surface plasmon-polariton waves guided by the interface of a metal and a rugate filter with sinusoidal refractive-index profile," J. Opt. Soc. Am. B, 27, 2218–2223 (2010).

ADHESIVE BOND INSPECTION FOR COMPOSITES WITH ULTRASONIC GUIDED WAVES

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Composite adhesive joint is increasingly used in aerospace industries. Ultrasonic guided wave techniques are regarded as promising methods for adhesive joint inspection. The advantages of using ultrasonic guided wave are summarized as: large area coverage, low weight added to the structure, large number of mode selection and no requirement of directly accessing the bonded region. To select effective modes for bond inspection, two guidelines are followed here, (1) Select the mode that is sensitive to the interface condition. (2) Select the mode excited in skin region that can be transformed to the mode in bonded region. [1,2]

Theoretical analysis

In wave propagating problem, the dispersion curves characterize the wave propagation in a given waveguide. Wave structures correspond to eigen vectors or mode shapes in a eigen value problem which descript the vibrating patterns of modes. Two challenges of inspecting adhesive bond are 1) finding a particular propagating mode shape with shear energy concentration at adherend-adhesive interface, and 2) exciting this propagating mode effectively by a mode conversion from the mode in waveguide A. By wave structure analysis, 2 mode-frequency combinations have been selected whose shear energy is concentrated at interface when the inplane displacement at that point is greater than elsewhere. Dispersion curves matching and wave structures matching promise an effective wave mode excitation at waveguide B. Two optimal

modes, mode 6 at 750 kHz and mode 7 at 950 kHz, and one counter example, mode 3 at 400 kHz, have been selected for further analysis.

Finite element simulation

The finite element simulation has been performed using ABAQUS to verify that the theoretical mode selection produces expected displacement. Either optimal mode produces displacement field such that the in-plane displacement at adherend-adhesive interface is greater. For the counter example, the in-plane displacement is not large at the interface.

Experimental results

The experiment has been done on two specimens with different sizes of adhesive defects. The adhesive defect is simulated by inserting Teflon film between adherend and adhesive layer during manufacturing. Ultrasonic guided wave is excited using angle beam wedge. Frequency content and amplitude ration have been selected as defect indicators. From the result of mode 6 at 750 kHz, it is found that frequency content changes with the presence of defects. For mode 7 at 950 kHz, amplitude ratio decreases evidently when defect exists. For the counter example, no obvious change is observed in terms of either frequency content or amplitude ratio.

 Rose, J. L. (1999). "Ultrasonic Waves in Solid Media." <u>Cambridge University Press</u>.
 Rokhlin, S. I. (1991). "Lamb wave interaction with lap-shear adhesive joints: theory and experiment." <u>Journal of the Acoustical</u> <u>Society of America</u> 89(Copyright 1991, IEE): 2758-2765.



Sketch of the structure adhesive bond, skin and stringer are 16-layer carbon-fiber-reinforced polymer with stacking sequence of $[0/45/90/-45]_{s2}$, adhesive layer is epoxy





Wave structures on dispersion curves of waveguide A and B



Phase velocity dispersion curves for waveguide A and B



The experiment setup, two angle beam wedge on a CFRP plate



Matching coefficient plot on dispersion curves



Finite element simulation result



Experiment results for adhesive defect detection

ESM TODAY SOUND DRIVEN SOFT MICROSWIMMERS

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Fundamental propulsion mechanisms at macro, micro and nano scale have received enormous attention both in the physics, chemistry and engineering communities. Thus far self-fueled or externally controlled propulsive systems are used in mixing, load transportation, etc. However, the potential of these microrobots lie in revolutionizing numerous aspects of medicine, such as, drug¹ or radioactive source² to a tumor cite in a body, microswimmers can act as occlusions to intentionally clog a blood vessel to inhibit tumor recirculation³, etc. Here we present a novel microswimmer, propelled by an oscillating bubble in the presence of sound waves. We propose a system that allows maneuvering each swimmer independently with capabilities of moving along straight and curved paths.

Soft microswimmers are fabricated by infusing poly (ethylene glycol) (PEG) containing photosensitive initiator inside the microfluidic channel. Hollow cone inscribed in cylindrical shaped particles, shown in Fig 1, are formed by exposing the flowing oligomer for 0.2 s to Ultraviolet (UV) light via an array of ring-shaped transparency mask. The mask is attached to the field stop of an inverted microscope. The structures are then washed in ethanol solution to remove the unpolymerized PEG in the conical section of the cylindrical particles. The particles were then treated with 1H,1H,2H,2H-perfluorooctyltrichlorosilane to make them hydrophobic, allowing a repeatable bubble to be trapped in the conical section when immersed in liquid, Fig 2a.

A droplet of liquid containing these soft microswimmers is transferred to a glass slide containing a piezo transducer. In the presence of sound field, the bubble oscillates transversely, in and out within the opening of the conic section. The operating frequency of the transducer is set to that of the natural frequency of the bubble, for maximum amplitude oscillation. Image sequence taken by highspeed recording at 225,000 frames /s shows the oscillation of bubble over one cycle, Fig. 2 b-i. The

recordings exhibit the bubble displacement as a function of time which is fitted to a sine; the driving frequency is 28.1 kHz, Fig. 2j. During oscillation, frictional forces are developed between the boundary of the bubble and the surrounding medium, resulting in a pressure gradient in the fluid giving rise to a bulk fluid flow, results in prominent recirculating flow regions shown in Fig. 2k. This phenomenon is commonly referred to as acoustic "microstreaming"⁴. Fig. 21 shows the initial position of the PEG swimmer in the absence of sound waves. As the amplitude of the sound field is gradually increased, the stationary soft swimmer transforms into a selfpropelled swimmer, Fig 2m. Fig. 3 shows x and y displacement of the swimmers over time. Direction of each swimmer is determined by their initial orientation and then propel in straight trajectories unless it comes in close proximity of another swimmer with different size of bubble trap. In that situation, secondary Bjerknes forces become prominent. As a result, the swimmer is either gets attracted or stops moving or its path gets deflected by the repulsive force of the neighboring swimmer. Though the velocity of the swimmers can be tuned by the strength of the applied sound while visually monitoring the oscillation amplitude for a distinct peak. Now by exciting each swimmer based on the different sized bubble trap, one can independently propel each swimmer. Though, the concept of specificity in terms of propulsion is proposed, manoeuvring each swimmer to a desired path is still a necessity.

We believe that these swimmers will find themselves widely useful in numerous applications ranging from microfluidics such as selective mixing, pumping, and load transportation at any specific cite to noninvasive medical surgical needs such as targeted drug delivery, brachytherapy², ablation, etc.

Word Counts: 497

References

1. Dong LX, Nelson BJ. 2007. Robotics in the small. Part II: Nanorobotics. IEEE Robot. Autom. Mag. 14(3): 111-212 2. Devlin PM. 2007. Brachytherapy: Applications and Techniques. Philadelphia: Lippincott Williams & Wilkins, 448 pp.

3. Nelson BJ, Kaliakatsos LK and Abbott JJ. 2010. Microrobots for minimally invasive medicine. Annu. Rev. Biomed. Eng. 12: 55-85

4. Leighton TG. 1994. The Acoustic Bubble. Academic Press: London.



group of soft swimmers.



Fig. 3 X and y displacement of swimmer's path

Δt = 0.37s а Displacement b 20 µm 60 µm j k Off t = 0 s Displacement [µm] **Final position** m 0 -2 -8 150 120 180 210 240 0 30 60 90 30 µm 100 µm On Time [µs]

Fig. 3 a. A PEG swimmer with a bubble trapped in the conical section of the structure. **b-i** Image sequence taken by high-speed recording at 225,000 frames /s representing the oscillation of bubble over one cycle. **j**. Bubble displacement as a function of time which is fitted to a sine. **k**. Experimental observation of microstreaming effect on particles during bubble oscillation. **l**. Initial position of the swimmer, when the sound wave is turned off. **J**. The swimmer starts to propel in the same trajectory of the initial orientation.

SPACE CHARGE FORMATION AND HIGH FIELD PROPERTIES OF LOW ALKALI GLASSES

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One of the biggest engineering innovations in the 20th century was the development of capacitor, a solid-state power conversion device, which led to dramatic changes in the way electric power is stored and distributed. New technology in power electronic products like hybrid electric vehicles, implantable medical devices and high power microwave systems require ultra-capacitors with decreased volume, weight, cost and energy densities greater than 30 J/cm³. Recently, thin alkali free borosilicate glasses have shown promising trends to be used as high energy density material with energy density as high as 35 J/cm³ owing to their high dielectric breakdown strength (12 MV/cm). These glasses maintain low dielectric loss up to temperatures of 200°C which is critical for automotive applications with extreme environmental conditions. In addition, thin glass sheets are sufficiently flexible to be wound into a compact capacitor structure. The possibility of fabricating multilayer storage capacitors with this thin glass has created considerable interest in recent vears for potential application to DC-bus capacitors in electric vehicles. So far polypropylene is the most commonly used dielectric in hybrid vehicles but they have low relative permittivity (2.1), energy densities approaching 10 J/cm³, they occupy significant volume in the capacitors and have limited high temperature capabilities. If these polymer based capacitors are replaced by glass capacitors it would result in five times decrease in capacitor volume which would certainly have a significant impact on the growth of hybrid vehicles which have increased by ten fold over the last five years.

Therefore, this study focuses on understanding the complex phenomenon of dielectric breakdown by studying intrinsic material properties (electrical and thermal conductivity, dielectric constant, and Young's modulus), extrinsic microstructural features (surface roughness, second phases and inclusions, porosity) and experimental conditions. This would help in further enhancing the energy density of these glass based capacitors.

Breakdown studies have shown increase in breakdown strength with decreasing glass thickness. The characteristic electrical breakdown field strength increased from 400 MV/m to 1100 MV/m as the glass substrate thickness decreased from 58 µm to 5 µm, respectively. The low field breakdown events are associated with macroscopic imperfections and the higher breakdown strength events are attributed to intrinsic electrical breakdown. A Weibull modulus as high as 50 was reported for the intrinsic breakdown region for semicrystalline polyethylene as compare to ~ 100 for the glass studied here. A higher modulus of intrinsic breakdown is expected for glass compared to the semicrystalline polymer since glass has a more homogeneous microstructure, transport and dielectric properties. A greater homogeneity of the material should be reflected in a higher Weibull modulus in the case of intrinsic breakdown.

To further investigate structural changes resulting in intrinsic breakdown, the glass was poled under several voltage and temperature conditions. Thermoelectric treatment of these glasses under high voltage (500V-2000V) and temperature (250°C-550°C) leads to depletion layer formation beneath the anode due to migration of monovalent and divalent cations. Migration of positive cations from the depletion layer increases the resistance of that layer resulting in all the voltage dropping across the thin depleted layer. This enables the glass to support fields closer to its intrinsic breakdown strength. The thickness of this depletion layer was found to depend on mobile ion concentration in the glass, poling temperature and electric field and is limited by the intrinsic breakdown strength of the glass. Beyond this field, electronic conduction in the depleted layer results in eventual breakdown of the sample. TSDC measurements confirm migration of Na⁺ and Ba²⁺ cations from below the anode with activation energy of 0.8 eV and 1.7 eV for Na and Ba motion respectively. Low frequency impedance have also spectroscopy results confirmed inhomogeneity in a poled glass due to formation of alkali depleted space charge layer.

A combination of high intrinsic breakdown strength, high intrinsic Weibull modulus, hightemperature capability, low dielectric loss and graceful failure observed in thin glass samples all point to their potential use as energy storage device.



Figure 1: Current transient for a 50 mic AF45 under a DC bias of 1 kV at 50-350°C. Current stabilized after the formation of space charge layer.



Figure 2: TSDC plot for 50 µm thick AF45 glass poled under a DC bias of 2 kV for two hours at 550°C. P1 and P2 shows the peak associated with the relaxation of Na and Ba cations respectively.



Figure 3: Cole- Cole plot for 19 µm thick AF45 poled under AC fields of 700V RMS at 400°C-550°C.



Figure 4: RC equivalent circuit model for poled glass.

Reference

- N. J. Smith, B. Rangarajan, M. T. Lanagan, C. G. Pantano, Materials Letters 63 (2009) 1245-1248.
- [2] D. E. Carlson, K. W. Hang, G. F. Stockdale, J. Amer. Ceram. Soc. 57 (1974) 295-300.
- [3] K. Takizawa, J. Amer. Ceram. Soc. 61 (1978) 475-478.
- [4] K. B. Albaugh, D. H. Rasmussen, J. Amer. Ceram. Soc. 75 (1992) 2644-2648.
- [5] Akira Doi, J. of Mater. Sc. 22 (1987) 761-769.
- [6] N. J. Smith, Doctoral Dissertation (2011).
- [7] C. M. Osburn, E. J. Weitzman, J. Electrochem. Soc. 119 (1972) 603-609.
- [8] G. De Mey and W. De Wilde, Elsevier sequoia S.A, (1974) 301-304.
- [9] A.E.Owen, Phys. and Chem. of Glasses 7 (1976) 174-192.
- [10] P.A. Timson, P. Wickens, Elsevier Sequoia S.A. (1970) R55-R58.
- [11] A. Servini, A. K. Jonscher, Thin Solid Films, 3 (1969) 341-365.
- [12] Luciana F. Maia, Ana C.M. Rodrigues, Solid State Ionics 168 (2004) 87-92.
- [13] R. M. Catchings, J. Appl. Phys. 50 (1979) 2813-2816.
- [14] R.C.Buchanan And M.A.Zuegel, J. Amer. Ceram. Soc. 51 (1968) 28-32.
- [15] Bernhard Roling, Malcolm D. Ingram, Solid State Ionics 105 (1998) 47–53.

DISPERSION RELATIONS FOR FLUID INTERFACE TRAPPED ACROSS HORSESHOE STRUCTURES

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ABSTRACT

Recent developments in acoustofluidics have triggered the importance of employing HSS for generating gradient indexed profiles, microstreaming and mixing etc for biological studies. То achieve these phenomenon the excitation of the microfluidic system at the natural frequency of the liquid-gas interface is critical. In this paper, we derive the dispersion relation and natural frequencies of a non-viscous liquidgas interface trapped across horseshoe structure and then extend this result to determine the damping effects caused by a viscous liquid on the interface.

Problem formulation

Interface

The boundaries of the interface S(x, y, t) are fixed. The interface is assumed to take the shape of the HSS's open face, initially. We seek a solution, satisfying the initial and boundary conditions, shown in equation 1.

 $S_{mn}(x, y, t) = Q_{mn} \sin(k_x x) \sin(k_y y) \cos(wt)$ (1)

where, Q_{mn} is the amplitude, $k_x = \frac{m\pi}{a}$, $k_y = \frac{n\pi}{b}$ are wavenumbers in x, y directions, m, n are mode numbers. For a small perturbation, the variations in pressure are balanced across the interface (z =0) given in equation 2. v_z is velocity of interface, ψ and φ are velocity potential of regions 1 and 2 respectively.

$$\rho_2 \frac{\partial \varphi}{\partial t} - \rho_1 \frac{\partial \psi}{\partial t} - 2\eta \frac{\partial v_z}{\partial z} = \sigma (S_{xx} + S_{yy})$$
⁽²⁾

For liquid in region 1, imposing similar conditions as above and noting that wave numbers (k_x, k_y) do not change for a non-viscous interface. Impedance condition from equation 3, and equation 2 with zero viscosity, gives the frequency modes (f_{mn}) given in equation 3.

$$\frac{k_1}{k_2} = \frac{Z_2 \rho_1}{Z_1 \rho_2}$$
(3)

$$f_{mn} = \frac{1}{2} \sqrt{\frac{\sigma Z_2 \pi}{\rho_2 (Z_1 + Z_2)}} \left[\frac{m^2}{a^2} + \frac{n^2}{b^2} \right]^{3/4} \tag{4}$$

$$f = \frac{1}{2} \sqrt{\frac{\sigma\pi}{\rho_{eff}}} \left[\frac{1}{a^2} + \frac{1}{b^2}\right]^{3/4}$$
(7)

$$+ i \pi \frac{\eta}{\rho_{eff}} \left[\frac{1}{a^2} + \frac{1}{b^2} \right]$$

where $k_1 = \left[\left(\frac{m\pi}{a} \right)^2 + \left(\frac{n\pi}{b} \right)^2 \right]^{\frac{1}{2}}$, $\rho_{eff} = \frac{\rho_2(Z_1 + Z_2)}{2}$. The analysis presented here is of

 r_{Z_1} . The analysis presented here is of use in acousto-opto fluidics and acousto-fluidics. Some important results are shown in figures 1a,1b,2a,2b.

REFERENCES

- 1. L.D. Landau, E.M Lifshitz, Fluid Mechanics, 2nd edition, Elsevier, 1987.
- L. E Kinlser, A. R. Frey, A. B. Coppens, J. V. Sanders, Fundamentals of acoustics, 4th edition. john Wiley and sons, Inc., 1999.
- 3. J. C Earnshaw, A. C. McLaughlin, "Waves at liquid surfaces: coupled oscillators and mode mixing", *Proceedings: Mathematical and Physical Sciences*, Vol. 433, No. 1889, pp.663-678.
- 4. Peter Behroozia, "The calming effect of oil on water", 2007 American Association of Physics Teachers. *Am. J. Phys.* 75, 5 May 2007.
- 5. A. J. Pretlove, "Free vibrations of a rectangular panel backed by a close rectangular cavity", *J. Sound Vib* 1965, 2 (3), 197-209.

- J. Lucassen "Longitudinal capillary waves. Part 1.—Theory", *Trans. Faraday Soc.*, 1968, 64, 2221-2229. DOI: 10.1039/TF9686402221
- E. H. Lucassen- Reynders, D J. Lucassen, "Properties of Capillary Waves", Advances in Colloid and interfacial science. vol. 2, 347-395, 1969.
- 8. A. J. Pretlove, "Forces vibrations of a rectangular panel backed by a closed rectangular cavity", *J. Sound Vib.* 3 (3), 252-261, 1966.
- K. Y. Lee, T. Chou, D. S. Chung, E Mazur, "Direct Measurement of the Spatial Damping of Capillary Waves at Liquid-Vapor Interfaces", *J. Phys. Chem.* 97, 12876, 1993.





SENSORS FOR IN-PILE ULTRASONIC MEASUREMENTS OF FUEL MATERIAL EVOLUTION

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In the strategic plan for in-pile instrumentation development in the DOE Fuel Cycle Research and Development (FCR&D) program ultrasonics-based technology was identified as a key approach that should be pursued to obtain the high-fidelity, highaccuracy data required to characterize the behavior and performance of new candidate fuels during irradiation testing [1]. However, these programs are severely limited by the lack of existing knowledge of ultrasonic transducer durability under irradiation conditions. This presentation addresses progress made by the authors towards characterization of ultrasonic sensors for such applications.

Further motivation has been provided by the recent Fukushima disaster, which has again demonstrated the need for improvements in nuclear power technology. The Fukushima disaster was essentially unstoppable once initiated, however our work has the potential to help prevent such disasters. The inflection point in the course of events which lead to the numerous explosions began with the generation of hydrogen from the zirconium alloy cladding. This hydrogen is generated when the temperature exceeds specified limits, in this case due to the inability to pump coolant, and occurs with all metal cladding materials [2]. Herein lays a clear demonstration of the need for new cladding materials, the development of which will be greatly facilitated by ultrasonic sensors.

The most basic form of ultrasonic inspection, namely the A-scan, yields a wealth of data in a single waveform. These data include:

- Time of Flight
 - o Dimensional changes
 - Elastic moduli
 - Density
 - Temperature
- Non-Linear Parameter β
 - Micro-crack formation and growth
 - Dislocation density
- Ultrasonic Attenuation
 - Average grain size
 - Viscoelastic effects

This list essentially sells itself, although bringing such a method to fruition in a reactor environment has been delayed by the lack of a suitable ultrasonic sensor. Admittedly, development of sensors for specific applications and the corresponding data analysis is anything but trivial. However, the first step would appear to be the selection of a suitable piezoelectric material, as is our aim.

The selection of a suitable piezoelectric material for the generation of ultrasound begins by selecting materials which are stable up to 600° C. An evaluation of the nuclear cross sections, with particular attention being paid to transmutation reactions, is then conducted. Finally, the materials ability to recover from ballistic radiation damage and immunity to radiolysis is checked. As a result of this down selection process Aluminum Nitride (AIN) is identified as an excellent candidate material which is known in other fields to be a radiation tolerant material [3-6], and is capable of withstanding temperatures of 1000° C [7-8].

Although this material is considered radiation tolerant, the extent to which it can maintain transduction in a radiation environment has not been characterized until now. This was achieved by fabricating a radiation resistant transducer, Figure 1: (a), and designing an experiment for in-core measurements Figure 1: (b). In Figure 1 (a) Aluminum 6061(1) was screwed into the top housing (8) made of Aluminum and was grounded. A single crystal AlN disc (2) with a center frequency of 13.4 MHz was coupled to the sample by aluminum foil. A Carbon-Carbon backing (3) was used to eliminate reverberations and a Nickel plunger (4) was used to apply pressure to the piezoelectric element and to act as the electrical lead. Alumina (5 & 7) was used as electrical insulation between the outer casing and the AlN. In Figure 1: (b) over 50 ft low loss cable was used between the electronic equipment and the sample setup. A vector network analyzer was used to gather impedance data while a computer controlled pulse-echo excitation/acquisition as well as obtained and stored data.

In order to evaluate the performance of the piezoelectric material pulse-echo measurements were made in-situ. The peak-to-peak amplitude was used to evaluate the performance on a pass fail bases while the bandwidth was used to evaluate coupling parameters. It was found that transduction was not inhibited up to 1.85×10^{18} n_{fast}/cm² and 5.8×10^{18} n_{thermal}/cm², Figure 2 and Figure 3. Further, the clarity of the pulse-echo measurements allowed both the characterization of temperature as well as nonlinear behavior in wave guide. Because unstable isotopes generated through transmutations are have relatively short half-life's post irradiation analysis

was done in lab facilitating further high temperature measurements and characterization of the d_{33} parameter. The piezoelectric characteristics of the AlN remained unchanged. Further discussion of these results can be found in the following references [9] and [10].



Figure 1: (a) Radiation resistant transducer design for BNR insertion. (b) BNR experimental setup with computer to pulse/receive, control vector network analyzer (VNA)



Figure 2: Pulse-echo A-scan did not change much due to irradiation.



Figure 3: Absolute amplitude during irradiation remained stable indicating that transduction is unchanged.

References

 Rempe JL, H MacLean, R Schley, D Hurley, J Daw, S Taylor, J Smith, J Svoboda, D Kotter, D Knudson, M Guers, SC Wilkins and LJ Bond. (2011). Strategy for Developing New In-pile Instrumentation to Support Fuel Cycle Research *and Development.* INL/EXT-10-19149, Idaho National Laboratory, Idaho Falls, Idaho.

2. Pool, R. (2011). FUKUSHIMA: THE FACTS. Engineering & Technology, 112(4), 221-2. Retrieved from

http://www.ncbi.nlm.nih.gov/pubmed/21819007

- Ronning, C., Dalmer, M., Uhrmacher, M., Restle, M., Vetter, U., Ziegeler, L., Hofsäss, H., et al. (2000). Ion implanted dopants in GaN and AlN: Lattice sites, annealing behavior, and defect recovery. Journal of Applied Physics, 87(5), 2149. doi:10.1063/1.372154
- Kucheyev, S. O., Williams, J. S., Zou, J., Jagadish, C., Pophristic, M., Guo, S., Ferguson, I. T., et al. (2002). Ion-beam-produced damage and its stability in AlN films. Journal of Applied Physics, 92(7), 3554. doi:10.1063/1.1501746
- Atobe, K., Okada, M., & Nakagawa, M. (2000). Irradiation temperature dependence of defect formation of nitrides (A1N and c-BN) during neutron irradiations. Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, 166-167, 57-63. doi:10.1016/S0168-583X(99)01184-2
- Trachenko, K. (2004). Understanding resistance to amorphization by radiation damage. Parks, D. A., Tittmann, B. R., & Kropf, M. M. (2009). Aluminum Nitride as a High Temperature Transducer. *Quantatative Nondestructive Evaluation*, 1029-1034.
- Patel, N. D., & Nicholson, P. S. (1990). High Frequency - High Temperature Ultrasonic Transducers. NDT International, 262-266.
- Stubbs, D., & Dutton, R. (1996). High-Temperature Ultrasonic Sensor for in Situ Monitoring of Hot Isostatic Processing. SPIE, 164-172.
- 9. D A Parks, B R Tittmann and B T Reinhardt, "Piezoelectric Material for use in a Nuclear Reactor Core," in Review of Progress in Quantitative Nondestructive Evaluation, edited by D. O. Thompson and D. E. Chimenti, AIP Conference Proceedings, American Institute of Physics, Melville, NY, 2011.
- 10.B T Reinhardt, D A Parks and B R Tittmann, "Measurement of Irradiation Effects in Precipitate Hardened Aluminum Using Nonlinear Ultrasonic Piciples (in-Situ)," in Review of Progress in Quantitative Nondestructive Evaluation, edited by D. O. Thompson and D. E. Chimenti, AIP Conference Proceedings, American Institute of Physics, Melville, NY, 2011.

PCR-FREE VARIABLE NUMBER TANDEM REPEAT GENOTYPING WITH SAMPLE STACKING CE

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Abstract

Variable number tandem repeat (VNTR) typing has been widely applied to genetic analysis. The general approach for VNTR typing is PCR amplification at the specific VNTR region and separate the amplicons bv capillary electrophoresis (CE). Here we present a new method for VNTR typing, which adopts ligase reaction instead of PCR thermal cycling, magnetic bead based solid phase capture for purification, and an elongated sample stacking CE for highly sensitive fluorescence detection as shown in Figure 1. In this assay, fluorescently ligation labeled probes, which are complementary to a repeat unit, randomly hybridize with the minisatellite tandem repeats, followed with a ligation of the adjacent probes on the DNA templates. After the ligation reaction, a large amount of ligation products in equidifferent length corresponding to each repeat number are generated. As a result, the repeat number of VNTR can be obtained directly by counting the number of the fluorescent peaks after a µCE separation. In our study, we used synthetic DNA templates with one to four 16-bp repeat units for thermal-cycling and isothermal ligation reaction. The study of sensitivity and minimum ligation reaction time was also carried out. To improve the signal intensity during CE, we used a novel sample stacking CE method as shown in Figure 2, in which the ligation product occupied in the 5 mm stacking microchannel are preconcentrated efficiently in a defined narrow

region through the optimized sample stacking CE scheme, resulting in more than 8-fold improved fluorescence peak intensities compared with that of a conventional cross-injection μ CE method. Such signal enhancement allows VNTR typing to be carried out at a low concentration of tested sample. We could successfully identify the repeat number of a 4-repeat VNTR template using sample stacking CE with only 25 fmol of DNA template in total 5 min of ligation reaction as shown in Figure 3. The PCR-free VNTR typing combined with the sample stacking CE would provide a novel platform for genetic analysis with high speed, low cost, high sensitivity and portability.



Figure 1. Schematic of a PCR-free minisatellite VNTR typing.



Figure 2. Sample stacking operation on a double-T microchannel (left panel) and the corresponding realtime images $(20 \times)$ of fluorescent signal (right panel).



Figure 3. Electropherogram of isothermal based ligated products.





Figure 1: (a) A schematic of the microfluidic device and the simulation result of the focused reagent iso-surface. (b) Chemical reaction used to synthesis the TTF-Au hybrid structure. (c) Schematic of different structured obtained..

Figure 2: The SEM images of the particles fabricated by 3D focusing method. The scale bar size is 1 um.

REFERENCES:

- [1] A. Ciesielski, C.-A. Palma, M. Bonini, and P. Samorì, "Towards supramolecular engineering of functional nanomaterials: pre-programming multi-component 2D self-assembly at solid-liquid interfaces.," Advanced materials (Deerfield Beach, Fla.), vol. 22, no. 32, pp. 3506-20, Aug. 2010.
- [2] K. F. Jensen, "Microreaction engineering is small better?," Chemical Engineering Science, vol. 56, no. 2, pp. 293-303, Jan. 2001.
- [3] J. I. Park, A. Saffari, S. Kumar, A. Günther, and E. Kumacheva, "Microfluidic Synthesis of Polymer and Inorganic Particulate Materials," Annual Review of Materials Research, vol. 40, no. 1, pp. 415-443, Jun. 2010.
- [4] a Rozanski et al., "Shear-induced crystallization of isotactic polypropylene based nanocomposites with montmorillonite," European Polymer Journal, vol. 45, no. 1, pp. 88-101, Jan. 2009.
- [5] V. Génot et al., "Synthesis of organic nanoparticles in a 3D flow focusing microreactor," Chemical Engineering Journal, vol. 161, no. 1-2, pp. 234-239, Jul. 2010.
- [6] M. Rhee, P. M. Valencia, M. I. Rodriguez, R. S. Langer, O. C. Farokhzad, and R. Karnik, "3D HYDRODYNAMIC FOCUSING FOR CONFINED PRECIPITATION OF NANOPARTICLES WITHIN MICROFLUIDIC CHANNELS," Sensors And Actuators, no. October, pp. 992-994, 2010.

ESM TODAY 2011 Charging of Nanodisks to Induce Shifts in Localized Surface Plasmon Resonance using Low Temperature Argon Plasma

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Metal nanostructures have unique optical resonance properties and the study of these properties is known as plasmonics. [1] In the case of nanostructures confined in all three geometric directions, this effect is known as localized surface plasmon resonance (LSPR). [1] Such structures can be used as optical devices based on LSPR shifts induced by altering the disk's geometry, [2] its material, [3] the surrounding material, [4] or its electron density. [5-7] Previous, the electron density was tuned using electrochemical charging, which is a slow process taking several minutes to hours. In this study, we induce high speed shifts in the LSPR of an array of gold nanodisks through plasma mediated charging.

A gold nanodisk array was fabricated on a glass substrate using electron beam lithography and placed in a vacuum chamber. The disks have a height of 30 nm, a diameter of 120 nm, and a periodicity of 300 nm. The test setup is depicted by the schematic in Fig 1a and an SEM image of the nanodisk array is shown in Fig 1c. Two flat windows on the wall of the vacuum chamber allowed the probe light to transmit through the sample and be collected and analyzed with an optical spectrometer. The extinction spectrum of the array of nanodisks was monitored using the spectrometer while argon plasma was generated in the chamber. The argon plasma was generated using two ring shaped electrodes, a high frequency function generator and a dynamic impedance matching network. The extinction spectrums of the nanodisk array before and after excitation of plasma are shown in Fig. 2b. The peak of the extinction spectrum "blue shifts" to a lower wavelength as a result of generating plasma in the chamber. A curve was fit to the experimental data to determine the peak position. The initial peak position (λ_i) was 661.1 nm and the final peak position (λ_f) was 655.7 nm. Previous work has shown that a blue shift indicates a negative charge generated on the nanoparticles. [5-7]

The increase in charge density based on the wavelength shift can be predicted using: [7]

$$N_f = \frac{\lambda_i^2}{\lambda_f^2} N_i, \qquad (1)$$

where N_f and N_i are the final and initial electron density, respectively. Using $N_i = 5.9 \times 10^{28}$ electrons/m³ and the previously stated peak positions, we calculated $N_f = 5.9976 \times 10^{28}$ electrons/m³. Using the calculated N_{f_f} a DDA simulation was performed to approximate the extinction spectrum of the nanodisk array. The results of the DDA simulation are shown in Fig 2b. Despite the good agreement between the experiment and the simulation, Eqn. 1 is known to overestimate the electron density. [6]

"Low temperature" argon plasma contains high temperature electrons and room temperature argon ions. The high temperature electrons have higher velocities and collide with the gold nanodisks more frequently than the ions, as depicted in Fig. 1b. When an electron collides with the nanodisk, it remains of the surface, adding a negative charge to the nanodisk, and each ion collision removes and electron increasing the charge on the disk. This process eventually reaches equilibrium resulting in a net negative charge on the nanodisks.

The dynamics of our experiment was monitored by recording the extinction spectrum every 0.1 seconds while the plasma was generated. The variation of the peak position with time is shown in Fig 3 for three separate experiments. The variation of the peak position before generating the plasma was low, and immediately after generating the plasma the peak position quickly blue shifts. After some time the peak position settles at a particular value. The charging takes place in only a few seconds; however, the discharging process takes up to one week. After the first test, the plasma was removed but no shift in the peak position occurred. The plasma was reignited and nothing happened. The sample was left for 1 week in atmospheric conditions. At the end of the week the peak position was back to its initial state. Plasma was reignited in the chamber, and a similar blue shift was observed. The nanodisks were charged by the plasma but had not electrical path to ground for discharging. Eventually, the nanodisks discharged through the glass of the chamber. This study could be used to study the dynamics of the charging induced by plasma and the charge density relationship with nanostructures. Also, the high speed charging induced by the plasma could be used for optical switching. However, a fast discharging method is needed for all of these applications.



Figure 1. (a) Schematic of the experiment. (b) Charging and discharging effect on particles in plasma (c)SEM image of the nanodisk array



Figure 2. (a)Experimental Extinction spectrum before and after plasma generation. (b)DDA Simulation of Extinction Spectrum.



Figure 3. Dynamics of shift of the peal of the extinction curve for three separate tests.

Figure 4. Diffusion coefficient of $CaCl_2$ at various flow rates (a) and concentrations.

REFERENCES:

- [1] S. Maier, *Plasmonics: fundamentals and applications*. Springer Verlag, 2007.
- [2] Y. B. Zheng, L. Jensen, W. Yan, T. R. Walker, B. K. Juluri, L. Jensen, and T. J. Huang, "Chemically tuning the localized surface plasmon resonances of gold nanostructure arrays," *J. Phys. Chem. C*, vol. 113, no. 17, pp. 7019–7024, 2009.
- [3] J.-H. Kim, W. W. Bryan, and T. Randall Lee, "Preparation, characterization, and optical properties of gold, silver, and gold?silver alloy nanoshells having silica cores," *Langmuir*, vol. 24, no. 19, pp. 11147– 11152, 2008.
- [4] Y. B. Zheng, Y.-W. Yang, L. Jensen, L. Fang, B. K. Juluri, A. H. Flood, P. S. Weiss, J. F. Stoddart, and T. J. Huang, "Active molecular plasmonics: Controlling plasmon resonances with molecular switches," *Nano Lett.*, vol. 9, no. 2, pp. 819–825, 2009.
- [5] B. K. Juluri, Y. B. Zheng, D. Ahmed, L. Jensen, and T. J. Huang, "Effects of geometry and composition on charge-induced plasmonic shifts in gold nanoparticles," *J. Phys. Chem. C*, vol. 112, pp. 7309– 7317, May 2008.
- [6] A. C. Templeton, J. J. Pietron, R. W. Murray, and P. Mulvaney, "Solvent refractive index and core charge influences on the surface plasmon absorbance of alkanethiolate monolayer-protected gold clusters," *J. Phys. Chem. A*, vol. 104, no. 3, pp. 564–570, 2000.
- [7] T. Ung, M. Giersig, D. Dunstan, and P. Mulvaney, "Spectroelectrochemistry of colloidal silver," *Langmuir*, vol. 13, pp. 1773–1782, Mar. 1997.

ESM TODAY 2012 DROPLET-BASED FLUORESCENE DETECTION IN OPTOFLUIDIC DEVICE

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The droplet-based microfluidics emerged as a new and smart technology to replace the conventional pipettes, plates or plats for the chemical or biochemical study, because they allowed numerous pico- or nano-liter volume samples and reagents dispersed into the isolate droplets for operations and reactions[1-2]. According to the advantages of these well-defined laboratories such as minimal sample consumption, less cross-contamination, fast mixing, miniaturized space, and achievement of different detections, they were successfully used for a diverse range of applications including particles syntheses, protein crystallization, PCR reactions, enzyme reaction, cell assay and so on[2-6]. Although the droplet manipulation technology including droplet generation, transportation, separation, fusion, trapping and mixing are all well achieved, only a few detection technologies are available for the droplet contents analysis. The drawback of those detection method as Microcopy or integrated with fluorescence detection system, cofocal microscopy is expansive and not suitable for the point of care diagnosis [7-8]. The development of basic minimization detection instrument including low cost, high throughput assay capability and high sensitivity becomes a critical issue and draw increasing attentions.

Based on the "microfluidic drifting" and "integrated optical fibers to allow the coupling of laser detection light and on-chip detection of various optical signals" technique developed by our group [9-10], we have developed a simple on-chip optofluidic system for droplet fluorescence analysis of biochemistry samples. A single microfluidic channel with droplet generator, droplet mixing and detection function components was fabricated in PDMS. T-shape channels were design to product droplet of different reagents. Chamber induced droplet fusion structure and long mixing channel were made as the micro-reactor for the selective reaction. Guide channels were fabricated to align two optical fibers on one side of the microfluidic channel as shown in figure 1. The red and blue droplets are dispersed to the mineral oil flow and driven forward to the fusion region. Uniform droplet was obtained after passive mixing functional unit. In the droplet detection area, light of 488nm blue laser is projected from one optical fiber to the side of the microfluidic channel. The blue laser is excitated the fluorescence dye in the droplet and given out green fluorescence signal. Then the green fluorescence signal is received by the side fluorescence detection optical fiber. After filtering the light wave only around 532nm, the fluorescence signal is converted to digital signals, and then collected and recorded by the oscilloscope.

The fluorescence is the common detection method for the biochemistry reaction, biology assay or the clinical diagnosis. In the proof of the concept demonstration, the flourscein (an absorption maximum at 494 nm and emission maximum of 521 nm) was used to test the fluorescence signals. As such, fluorescent signal can be used to indicate the reaction inside the droplet. The uniform droplets were tested and recorded in figure 2a. The displayed voltages of the different droplet well defined the fluorescent intensity of the single droplet.

Then the 20uM fluorescein and water were injected into the reagent channels and maintained at the same flow rate at 0.5 ul/min and dispersed by the mineral oil with flow rate 2 ul/min as shown in figure 1(b). Then the 20uM fluorescein and water droplet with same volume were driven to the fusion channel and mixing well, and then detected as the figure 1(c) shown. The dark flied image described that the blue laser beam sharing on droplet and the green fluorescent light was excitanted and collected by the side wall detection fibers. The single droplet fluorescent signal was recorded as figure 3. Then voltage signal of the same volume ratio diluted 20uM fluorescein droplet was comparable to the only 10uM fluorescein droplet which indicated that our device has strong potential to be used to the quantification measurement. Later, the designed Probe and DNA single strain sample whose specific binding could have fluorescent signal could be achieved for cancer detection.

In conclusion, we have presented a compact packetable microfluidic device capable of generation, merging, mixing and detection fluorescence droplets. This device could be implemented in hand-held lab-on-a-chip systems to indicate fluorescence and related biochemistry reaction as DNA hybridization.



Figure 1. (a)Schematic optofluidic device for droplet fluorescence detection.(b) Two different droplets were produced from the T shape droplet generator.



Figure 2. Fluorescence detection signals of droplets with 10uM flourescein.



Figure 3. Fluorescence Signal of single droplet through the detection region. The green fluorescence light was excitated by the blue laser when the 10uM flourescein droplet went through the detection region.

REFERENCES:

- A. Theberge, F. Courtois, Y. Schaerli, M. Fischlechner, C. Abell, F. Hollfelder and W. Huck, Angew. Chem., Int. Ed.49, 5846 (2010).
- [2] H. Song, D. L. Chen and R. F. Ismagilov, Angew. Chem., Int. Ed. 45, 7336 (2006).
- [3] L. B. Zhao, L. Pan, K. Zhang, S. S. Guo, W. Liu, Y. Wang, Y. Chen, X. Z. Zhao and H. L. W. Chan, Lab Chip 9, 2981 (2009).
- [4] N. R. Beer, B. J. Hindson, E. K. Wheeler, S. B. Hall, K. A. Rose, I. M. Kennedy and B. W. Colston, Anal. Chem. 79, 8471 (2007).
- [5] M. He, J. S. Edgar, G. D. M. Jeffries, R. M. Lorenz, J. P. Shelby and D. T. Chiu, Anal. Chem. 77, 1539 (2005).
- [6] F. Guo, X. H. Ji, K. Liu, R. X. He, L. B. Zhao, Z. X. Guo, W. Liu, S. S. Guo and X. Z. Zhao, Appl. Phys. Lett. 96, 193701 (2010).
- [7] J. Clausell-Tormos, A. D. Griffiths and C. A. Merten, Lab Chip 10, 1302 (2010).
- [8] K. Churski, P. Korczyk and P. Garstecki, Lab Chip 10, 816 (2010).
- [9] Huang, H.; Mao, X.; Lin, S.-C. S.; Kiraly, B.; Huang, Y.; Huang, T. J. Lab Chip 2010, 10 (18), 2387-93.
- [10] Mao, X.; Lin, S.-C. S.; Dong, C.; Huang, T. J. Lab on a Chip 2009, 9 (11).

ESM TODAY GRADIENT PLATE FOR NOISE REDUCTION AND ENERGY HARVESTING

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The goal of this research is to incorporate phononic crystals within lightweight aerospace panels to reduce interior noise in rotorcraft vehicles. The strategy we took is developing a new type of panels that can focus and dissipate structuralborne energy. By taking advantage of the geometrydispersive nature of flexural bending waves, we can locally adjust the bending wave speed by changing the thickness of the plate to obtain a speed gradient across the wave propagation direction. We have designed and simulated two different gradient-index plates both can focus structureborne energy to a hot spot over relatively wide working frequency range. Both designs are sent out for fabrication now. The future work includes experimental verification and optimization of the designs. We also seek to extend the idea to address the airborne noise issue.

Word Count: 131



Figure 1: Flexural wave speed in air/aluminum PCs with different lattice periods. (left: along GX orientation, right: along GM orientation).



Figure 2: Simulation of wave propagating in the GRIN PC plate.

REFERENCES:

- [1] S.-C. S. Lin and T. J. Huang, J. Appl. Phys. 106, 053529 (2009).
- [2] S.-C. S. Lin, T. J. Huang, J.-H. Sun, and T.-T. Wu, Phys. Rev. B 79, 094302 (2009).
- [3] S.-C. S. Lin, B. R. Tittmann, J.-H. Sun, T.-T. Wu, and T. J. Huang, J. Phys. D: Appl. Phys. 42, 185502 (2009).

On-chip single-shot characterization of enzymatic reaction constants K_m and k_{cat} via an acoustic-driven millisecond bubble-mixer

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In this paper, we present a novel strategy for rapid characterizing enzyme constants K_m and k_{cat} in a microfluidic system. This channel design involves a bubble anchored in one horseshoe structure which could be stimulated by a piezoelectric transducer to generate vortices in the fluid. Thus, the enzyme and substrate could be mixed rapidly within 100 milliseconds by the vortices to yield the product of the reaction. Enzymatic reaction constants K_m and k_{cat} can therefore be obtained from the reaction rate curves for different concentrations of substrate while keeping the enzyme concentration unchanged. Parallel experiments is carried out by single-shot experiment which in concentration is changed by injecting flow rate. We studied the enzymatic reaction for β-galactosidase and its substrate (resorufin- β -D-galactopyranoside) to measure ite reaction kinetic parameters.

The PDMS channel design was demonstrated in Fig. 1. The channel width, depth, and length are 570 μ m, 65 μ m, and 1.5 cm, respectively. A horseshoe structure is positioned at the center of the channel to trap a bubble inside (insert in Fig. 1). One bubble was trapped inside the horseshoe structure by injecting liquid from the outlet of channel. A piezoelectric transducer (Model No. 273-073, Radioshack Corp.) was bonded adjacent to the PDMS microfluidic device and driven by a function generator (Hewlett Packard 8116A) using a square wave with a frequency equal to the natural resonance frequency (31 kHz)

of the trapped bubble.

The enzyme for characterizing its reaction constant was β -galactosidase (from *E. Coli*). While the enzyme and substrate do not emit fluorescence, the enzymatic reaction is a fluorescence generating reaction. The reaction product, resorufin, has a maximum emitting fluorescent intensity at 585 nm, while the maximum exciting wavelength is 570 nm. A fluorescent intensity detection system was conducted to study the product concentration change. A microscope (Elipse Ti, Nikon Corp.) combined with two bandpass filters (530-570 nm and 580-650 nm, respectively) and one dichroic mirror (transmission above 575 nm) in a reflective mode was used to observe the fluorescent light.

The enzyme reaction rate could be calculated by the fluorescent intensity change of product. Then Lineweaver-Burk plot can be got by taking the reciprocal of initial reaction rate and substrate concentrations, using the linear regression analysis. we calculated the slope of the fitting line, which is equivalent to (K_m/V_{max}) , and the y-intercept, which was $(1/V_{max})$, k_{cat} can be calculated by V_{max} and enzyme concentration. In this reaction, K_m and k_{cat} are $483\pm183 \mu$ M and $73\pm26 1$ /s respectively, which are in accordance with the published data. The rapid mixing inherent in our system provides a solution for high-speed enzyme reaction kinetics studies and milliseconds to seconds level chemical reactions.



Fig. 1 Schematic figure of enzyme reaction channel and optical detection system. The inset shows the horseshoe structure with a bubble inside.



Fig. 2 (a) Enzyme reaction carried out in the channel, the fluorescent intensity was detected in the channel with equal distance intervals. (b) The fluorescent intensity at the beginning point of reaction (circle) and the point after a certain reaction time (square) The uniformed fluorescent intensity at the middle of the curves were used as effect fluorescent intensity in Fig. 2c. (c) Initial reaction rate (v_0) curve with substrate concentration ranged from 91 μ M to 545 μ M. The concentration of enzyme was set as 5 mg/l.



Fig. 5 Lineweaver–Burk plot to characterize K_m and k_{cat}

ENCAPSULATION OF POLYSTYRENE BEADS/CELLS IN AQUEOUS-IN-OIL DROPLETS

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Droplet-based microfluidic systems involve formation and manipulation of nano-/pico-liter scale droplet of one fluid phase in another immiscible fluid phase, such as water-in-oil or oil-in-water droplets. This droplet microfluidics is compatible with various biological researches since the formed droplets can serve as isolated reactor or carrier if we encapsulate different biological reagents inside the droplets, such as DNA, [1] proteins, [2], cells [3-5] and even organisms. [6] The droplet microfluidics system has some beneficial characters, such as high-throughput droplet generations, isolation inside individual droplets and compatibility with various downstream droplet manipulations, which make it advantageous over traditional methods in some research areas.

For some cell-based biological assays, small sample assumption, high-throughput, single cell level manipulation/detection are desirable, making cell encapsulation in droplets studies quite interesting. Suppose we can successfully capture single cell inside individual droplet, one challenge remains to be solved, which is how to separate cell-containing droplets from empty droplets or even how to separate droplets containing big cells from droplets containing small cells. One hydrodynamic method has already been developed to sort big droplets with cell encapsulated from small, empty droplets. [5] Inspired by this research, we are trying to develop a hydrodynamic method that can separate big cell-containing droplets from small cell-containing droplets. In generating the aqueous-in-oil droplets, the flow rates of aqueous phase and oil phase are controlled so that empty droplet sizes are comparable to cells (<15 μ m in diameter). In this situation, the encapsulation of big polystyrene beads/cells will generate droplets larger than those generated with encapsulation of small polystyrene beads/cells. Therefore, these droplets of different sizes can be separated hydrodynamically.



Fig.1 Encapsulation of single cells in aqueous-inwater droplets. Yellow arrows in Fig.1A show encapsulated single A549 cells in droplets. Green arrows in sequences of A-B-C-D show the encapsulation process of a single cell.



Fig.2 Encapsulations of small and big polystyrene beads generate droplets of different sizes. A & B show that encapsulation of 5 μ m PS bead generates droplet of the same size to empty droplets. C & D show that encapsulation of 10 μ m PS bead generates droplet larger than empty droplets (green arrow in D).



Fig.3 Fast camera images of decreasing droplet size to less than 15 μ m in diameter. In Fig.3A, the droplet size is too big to show the effect of bead encapsulation. In Fig. 3B, the droplet size is reduced to less than 15 μ m.

REFERENCES:

[1] P. Kumaresan, C. J. Yang, S. A. Cronier, R. G. Blazej and R. A. Mathies. High-throughput single copy DNA amplification and cell analysis in engineered nanoliter droplets. *Anal. Chem.* 2008, 80, 3522-2529.

[2] H. N. Joensson and H. Andersson-Svahn. Droplet microfluidics-a tool for protein engineering and analysis. *Lab Chip*, 2011, 11, 4144-4147.

[3] J.-C. Baret *et al.* Fluorescence-activated droplet sorting (FADS): efficient microfluidic cell sorting based on enzymatic activity. *Lab Chip*, 2009, 9, 1850-1858.

[4] J. Clausell-Tormos *et al*. Droplet-based microfluidic platforms for the encapsulation and screening of mammalian cells and multicellular organisms. *Chemistry & Biology*, 2008, 15, 427-437.

[5] M. Chabert and J.-L. Viovy. Microfluidic highthroughput encapsulation and hydrodynamic selfsorting of single cells. *PNAS*, 2008, 105, 3191-3196.

[6] W. Shi, J. Qin, N. Ye and B. Lin. Droplet-based microfluidic system for individual *Caenorhabditis elegans* assay. *Lab Chip*, 2008, 8, 1432-1435.

SUBMICRON SIZED THREE DIMENSIONAL HYDRODYNAMIC FOCUSING

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The usefulness of the flow cytometer lies in its extraordinary capability to detect single cells at very high speed. Our flow cytometry device is a microfluidic-based, single-layered, onchip device that is capable of performing cell/particle focusing via the hydrodynamic focusing phenomenon. The effectiveness of the flow cytometer can only be realized if the cells/particles can pass, one-by-one, through a particular point called "the interrogation point." Commercial flow cytometry devices achieve this by passing fluid containing the cells/particles through a small tube that has large concentric outer tubing through which the sheath fluid flows. Increasing the sheath flow rate increases the tightening of the core flow. Sheath fluids are extremely expensive and mainly contribute to the increasingly high running cost inherent with present flow cytometers. This increase in cost greatly affects the accessibility of this device to the masses as a routine test, and thus a higher rate of death occurs due to lack of sufficiently expedient diagnoses of victims. Therefore, it is urgently imperative this problem be overcome in order to substantially increase efficiency and quality of medical care, and in doing so, increase overall quality of human life.

A computational and experimental optimization of 3D hydrodynamic focusing via curved microfluidic channel has been achieved in this study. Curvature lengths of 90° , 135° and 180° forming angled curved path in microfluidic channels have been used to achieve tighter focusing width and the physical phenomenon governing this achievement have been discussed in this paper. We have shown that with increase in the curvature length greater than 90 degrees, it is possible to achieve a focusing width of 7 microns with 135° curvature length and up to as small as 1.5 micron focusing width with curvature length of 180° . Moreover, with such an increase in the vertical sheath in order of obtains the above mentioned focusing width has been proven with simulation results and demonstrated experimentally in this study.

$$De = \frac{\rho V D}{\mu} \left(\frac{D}{2R}\right)^{1/2} \tag{1}$$

where

- ρ is the density of the fluid
- μ is the dynamic viscosity
- V is the axial velocity scale

- D is the diameter (other shapes are represented by an equivalent diameter)
- R is the radius of curvature of the path of the channel.

. We have invented a newly engineered microfluidic-based device that will have extremely high throughput and much lower running cost without compromising the results, the quality of which depends on the focusing width of the flow cytometry device. In consideration of life threatening diseases, our device has the potential to perform the identification of cells affected by HIV, cancerous tumor cells, and the like. In this particular device we have demonstrated that our improved design has the potential for greater focus, and in turn, a higher achievability of detection for extremely small microorganisms comparable to the typical size of bacteria, which is around 1 micron. Moreover, in-depth knowledge of fluid mechanics and optimization through simulations has allowed for effective practical testing of the device, which further validates the claims made. By our invented methods we have been able to reduce the total flow rate by 50% and vertical sheath flow by more than 76%. Moreover, we provide herein, complete guidelines for designing principles for single-layered, onchip, hydrodynamic-based flow cytometry chips. By following the subsequent guidelines, and by extrapolating from the graphs presented in this document, flow cytometry devices can be build that will far surpass the performance of the commercial flow cytometer.

Word Count: 572



Figure 1:Simulation results of main channel cross-section for vertical sheath flow of 337.5 μ l/min at (a) 900 (b) 1350 (c) 1800 . Curvature length greater than 900 results in remixing of the sample flow.



Figure 2:(a)900 curvature showing ability to focus particles as small as 15 μ m (b)1350 curvature device with ability to focus as small as 7 μ m particles (c) 1800 curvature device indicating its ability to focus particles as small as 1.5 μ m.

REFERENCES:

- W. Inglis, J. A. Davis, T. J. Zieziulewicz, D. A. Lawrence, R. H. Austin and J. C. Sturm, J. Immuno. Methods, 329, 151, (2008).
- [2] D. W. Inglis, K. J. Morton, J. A. Davis, T. J. Zieziulewicz, D. A. Lawrence, R. H. Austin and J. C. Sturm, Lab Chip, 8, 925 (2008).
- [3] O. D. Laerum, T. Farsund, Cytometry Part B, 2, 1 (1981).
- [4] H. M. Shapiro, R. C. Leif. Practical flow cytometry: Wiley-Liss New York, 2003.
- [5] M. Brown, C. Wittwer, Clin. Chem., 46, 1221 (2000).
- [6] M. Rosenauer, W. Buchegger, I. Finoulst, P. Verhaert and M. Vellekoop, Microfluid. Nanofluid., 10, 761 (2011).

On-chip single *C. elegans* **manipulation and cells separation and sorting using tunable surface acoustic wave**

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Non-invasive cellular-scale manipulation is essential to many fundamental and biomedical studies. For example, the ability to precisely control the physical location of a cell facilitates the investigation of cell-cell and cell-environment interactions. This on-chip physical manipulation could also provide a tool to help researchers observe the behaviour and neuronal activity of organisms such as C. elegans (Caenorhabditis elegans) in a well-controlled environment. These tweezers might also aid in molecular dynamics/mechanics studies allowing by researchers to precisely control the physical location of a particle/particles conjugated with a biomolecule. Among the conventional manipulation techniques-including optical tweezers $(OT)^1$, optoelectronic tweezers $(OET)^2$, electrokinetic forces³, magnetic tweezers⁴, and acoustic traps⁵ -optical tweezers and DEP based optoelectronic tweezers demonstrate the ability to manipulate neutral particles dynamically and freely. They, both of which integrate complex optical elements, can achieve high resolution and high throughput, and offer great flexibility for trapping single particles. However, both of the two micromanipulation techniques cannot achieve high resolution, high throughput, and at the same time, low cost and portability. Here we present a surface acoustic wave based technique that can be used to control the dynamics of cells and organisms precisely by tuning acoustic frequency. It offers high resolution and high throughput, mean while simplicity, miniaturization and low cost as well. By controlling the frequency, thus the wave length of standing surface acoustic wave, the position of trapped particles can be controlled to achieve multiple manipulation functions without integrating optical components, which may increase the cost and size.

Ultrasonic wave has been well studied since hundred years ago, and has been so far a safe detection method widely used in biomedical and clinical medicine. We demonstrate dexterous manipulation of biological objects including cells and entire organisms (*C. elegans*). *C. elegans* is an attractive model organism for many biological and medical studies, mainly because of its relatively small size (~1 mm long), optical transparency, well-mapped neuronal system, diverse repertoire of behavioural outputs, and genetic similarities to vertebrates. However, trapping and manipulating *C. elegans* have proven to be difficult and may involve anaesthetics, vacuum, cooling, or directcontact mechanical procedures²⁵⁻²⁸. To our knowledge, our acoustic tweezers are the first to achieve contact-free, non-invasive, and precise manipulation/stretching of *C. elegans*. Cell separation and sorting are of broad importance in applications such as biomedical diagnosis in hospitals and environmental monitoring. Using our techniques, we successfully demonstrate the functions such as: blood cells separation from plasma, cells median exchange, and blood wash. We also achieve the Human white blood cell sorting into five different outlets, which means we can simultaneously sort five different types of cells in a single chip.

Our SAW based tweezers technique utilizes the tuning of frequency to control the position of trapped particles regardless of their properties of polarizability, optics, and magnetics. Compare with optical tweezers and optoelectronic tweezerds, our technique is low cost, simply structured, and low power. Figure 1 is the device sample for *C. elegans* manipulation. As shown in figure 2, *C. elegans* can be precisely controlled in x and y direction by tuning the frequency, and actively stretched in a noninvasive and contactless approach.

Word count: 514

Reference:

- [1] Grier, D. G. A revolution in optical manipulation. Nature 424, 810–816 (2003).
- [2] Pei Yu Chiou, Aaron T. Ohta & Ming C. Wu, Massively parallel manipulation of single cells and microparticles using optical images, NATURE, 436, 370-372 (2005).
- [3] Kremser, L., Blaas, D. & Kenndler, E. Capillary electrophoresis of biological particles: Viruses, bacteria, and eukaryotic cells. Electrophoresis 25, 2282–2291 (2004).
- [4] Yan, J., Skoko, D. & Marko, J. F. Near-fieldmagnetic-tweezer manipulation of single DNA molecules. Phys. Rev. E 70, 011905 (2004).
- [5] Jinjie Shi, Daniel Ahmed, Xiaole Mao, Sz-Chin Steven Lin, and Tony Jun Huang, Acoustic Tweezers: Patterning Cells and Microparticles Using Standing Surface Acoustic Waves (SSAW), Lab on a Chip, 9, 2890-2895, (2009)



Fig.1 Device sample consists of four interdigital transducers(IDTs). One pair of IDTs control the movement of particles in X direction while another pair in Y direction.



Fig. 2 Single *C. elegans* manipulation. One single *C. elegans* was (a) trapped, (b) moved in y direction, (c) moved in x direction, and (d) moved in y direction again and released, with the average velocity of ~40 μ m/s. *C. elegans* before (e) and after (f) being fully stretched..

(Poster and presentation)

PULSATILE GRADIENT GENERATION VIA ACOUSTIC STREAMING OF BUBBLES TO STUDY THE CHEMOTAXIS OF HL-60 NEUTROPHIL-LIKE CELLS

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Gradients of biochemical solutions are important in various biological processes. The purpose of microfluidic biochemical gradients is to regulate the microenvironment of a cell during controlled in vitro conditions, in order to emulate the cell's actual in vivo setting [1]. The spatiodynamic and temporal nature of various biochemical gradients can have a substantial effect on the chemotaxis of various cells and microorganisms. Because chemotaxis plays an important role in processes such as neutrophil migration, microbial processes, biofilm formation, disease pathogenesis, and contaminant bioremediation, it is a widely studied field of research today. Biochemical gradients also influence the movements of cells in processes such as gastrulation, organogenesis, and cancer metastasis.

Recent studies have shown that both magnitude and timing of chemical stimulus play an important role in cell behavior. With the foundation of previous publication [2], we have developed an active microfluidic gradient generator capable of generating both stable and pulsatile gradients. This device incorporates a microbubble trapped in a horse-shoe structure. When acoustically driven, the membranes (liquid/air interfaces) of these trapped bubbles start to oscillate. The bubble oscillation results in a microstreaming phenomenon-strong pressure and velocity fluctuations in the bulk liquid, thus giving rise to fast and homogenized mixing of two fluids flowing side-by-side. Acoustically manipulating the bubbles using a transducer connected to a function generator allows both stable and pulsatile gradient generation. Furthermore, the oscillation of the bubbles can be tuned directly, allowing various dynamic chemical profiles. To achieve a pulsatile gradient, five bubble structures are arranged in a ladder formation. By mixing and merging fluid along and across the channel, a stable gradient profile can be established. The performance of the gradient generator was characterized by FITCdextran and PBS solution. First order exponential decay gradients and other pulsatile profiles were investigated along with their stability.

For the cell study, this spatiotemporal gradient generator could potentially allow further investigation of HL-60 cell chemotaxis. The migration velocity, the response frequency and the turnover rate of HL-60 cells will be characterized.

REFERENCES:

- N. L. Jeon, H. Baskaran, Stephan K. W. Dertinger, George M. Whitesides, L. V. D. Water, M. Toner, "neutrophil chemotaxis in linear and complex gradient of interleukin-8 formed in a microfabricated device", *Nat. Biotechnol.*, vol. 20, pp. 826-830, 2002.
- [2] D. Ahmed, X. Mao, J. Shi, B. K. Juluri and Tony Jun Huang, "A millisecond micromixer via single-bubblebased acoustic streaming", *Lab. Chip.*, 9, pp. 2738-2741, 2009.



Figure 1 Schematic of the experimental setup, the piezo transducer (not shown) is placed adjacent to the microfluidic device and the generation of FITC-dextran gradient by sequential mixing of the bubbles.



Figure 2 (a) & (b) Images to show the trapping of the bubbles. (c) Image sequence to capture one complete oscillation of single bubble trapped in a horse-shoe structure by fast camera at a rate of 200,000 fps. (d) Image to show the homogenous mixing of FITCdextran by single bubble.



Figure 3 Characterization of gradient profile 1 inside different part of the microfluidic channel.



Figure 4 Characterization of other gradient profiles inside the microfluidic channel.

NITRIDE FORMATION VIA LASER-SUSTAINED PLASMA INTERACTION WITHOUT DIRECT IRRADIATION OF THE SUBSTRATE

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Industries ranging from aerospace to biomedical utilize titanium and its alloys due to their superior strength, high strength-to-weight ratio, low density, high melting point, excellent corrosion resistance, high fracture toughness, good heat transfer properties, and biocompatibility [1, 2]The poor tribological characteristics of these metals, however, reduce their applicability under severe wear conditions [3]. To enhance these properties, titanium nitride (TiN) coatings are often employed due to their extreme hardness, excellent corrosion and wear resistances, high thermal conductivity, important transport properties, chemical inertness, and gold appearance [4]. TiN is commonly used for protective coatings on cutting tools and drill bits, diffusion barriers in microelectronics, metal smelting crucibles, optical coatings, and decorative features [5].

Laser nitriding is a process in which laser irradiation melts a titanium surface while under a nitrogen-containing atmosphere. It has been investigated since the 1980s because of its potential as a fast, effective TiN synthesis techn1ique [6]. During laser nitriding, the interaction of the laser melt and species evaporated from the surface produces near-surface plasma. The impact of this laser-induced plasma is a subject of much discussion; some consider it to enhance the coating process [7-9], others say it is detrimental to nitridation [10, 11], while most ignore its potential effects [12-14]. The research to be presented investigates the effect of plasma on a Ti surface without direct laser melting by utilizing laser-sustained plasma (LSP).

LSP, originally referred to as a "continuous optical discharge" is plasma generated by a laser beam in a gaseous atmosphere that can be sustained indefinitely away from any potentially interacting surface [15]. While LSP has been successfully used to deposit diamond [16-18], its potential contributions to the formation of a broad range of hard coating compositions has not been systematically explored. LSP is an optical discharge plasma, similar to a laser-induced plasma. While this optical discharge plasma can be used to melt a surface, it also provides a novel method for studying the influence of plasma on a Ti substrate without the laser beam interacting directly with the surface.

There is an important distinction between the experimental process being presented and conventional laser nitriding. In laser nitriding, the main heat source responsible for melting is the laser beam. In the process under consideration, LSP is the heat source responsible for melting the titanium substrate. The term LSP nitriding is used to indicate that LSP, and not the laser, is being used at the heat source. The laser is used only to maintain the LSP and does not have any direct interaction with the material being nitrided.

In order to melt the surface without irradiating it, the LSP is translated to a location some distance, d, from the substrate, as shown in Figure 1. In all cases, it was determined there was no effect from the laser beam on the substrate without the LSP being present. Three values for d were tested: 2 mm, 2.5 mm, and 3 mm. Each distance produced a different melt pattern, as shown in Figure 2. These tests were performed on ASTM grade 2, commercially pure (< 99.5%) titanium, which was sectioned into 25.4 mm x 50.8 mm x 3.175 mm plates. Some were processed with the as-received, unpolished, milled surface, while others were polished mechanically prior to LSP exposure. In addition, length of exposure was considered.

During processing, images were collected via a CCD camera with appropriate filters to minimize oversaturation and modified frame grabbing software, so processing could be visually monitored. Scanning electron microscopy (SEM) with a backscattered electron (BSE) detector and energydispersive x-ray spectroscopy (EDS) were utilized to analyze the initial products of the process, as well as transverse and longitudinal metallographic cross-sections.

Using EDS results in conjunction with the macroscale images seen in Figure 3 and previous literature on nitriding, it was found that hypo-stoichiometric, near-stoichiometric [19], and hyper-stoichiometric [20] TiN developed at the main impact site. Also present were regions of TiO_2 [21] and locations where a nanoscale layer of TiO_2 covered a deeper layer of TiN [22].

Surface morphologies varied widely between the three values for *d* tested. d = 3 mm showed little structure, while the d = 2 mm and d = 2.5 mm samples showed more mature crystallization and evidence of evaporative growth. Crosssections showed solid and dendritic layers very similar to those seen in laser nitrided TiN. It was also noted that convection assists the permeation of nitrogen into the titanium melt, similar to the convective currents found in laser nitriding.

The regions of high nitrogen concentration, the convective movement within the melt pool, and the surface growth are the most significant structures seen in the LSP nitrided Ti. The faceted crystal growth on the surface and the dendritic layer below it suggest that there are two growth mechanisms at work during LSP nitriding: one in the melt and another from the vapor.

Word Count: 816



Figure 1: A sideview of the experimental setup showing d, the distance between the laser centerline (indicated by the green line) and substrate surface. The size and shape of the LSP are shown schematically. The green arrow indicates the direction of the laser beam, while the white, dashed line indicates the location of the laser's focal plane.



Figure 2: Macroscale images for the LSP parallel configuration after 5 seconds at: a) d = 2 mm; b) d = 2.5 mm; and c) d = 3 mm.

- Caron RN and Staley JT. Titanium and Titanium Alloys, in: Dieter GE (Ed.). ASM Handbook, vol. 20. Materials Park, OH: ASM International; 1997.
- [2] Zhang J, Fan D, Sun Y, Zheng Y. Key Eng Mater 2007; 353-358:1745.
- [3] Guo B, Zhou J, Zhang S, Huidi Z, Pu Y, Chen J. Mater Sci Eng A 2008;480:404.
- [4] Tavares J, Coulombe S, Meunier J-L. J Phys D: Appl Phys 2009;42:1020011.
- [5] Wu M. J Alloys Compd 2009; 486:223.
- [6] Spies H-J. Surf Eng 2010; 26:126.

- [7] Akarapu RK, Dua P, Campbell A, Scott D, Nassar A, Todd JA, Copley SM. Proc Mater Res Soc Symp 2007; 1040:Q08.
- [8] Höche D, Rapin G, Kaspar J, Shinn M, Schaaf P. Appl Surf Sci 2007; 253:8041.
- [9] Thomann AL, Sicard E, Boulmer-Leborgne C, Vivien C, Hermann J, Andreazza-Vignolle C, Andreazza P, Meneau C. Surf Coat Technol 1997; 97:448.
- [10] Chen X, Wu G, Wang R, Guo W, Yang J, Cao S, Wang Y, Han W. Surf Coat Technol 2007; 201:4843.
- [11] Kloosterman AB. Surface Modification of Titanium with Lasers. Groningen: Groningen University Press; 1998.
- [12] Jianglong L, Qiquan L, Zhirong Z. Surf Coat Technol 1993; 57:191.
- [13] Raaif M, El-Hossary FM, Negm NZ, Khalil SM, Kolitsch A, Höche D, Kaspar J, Mändl S, Schaaf P. J Phys D: Appl Phys 2008; 41:085208.
- [14] Mridna S, Baker TN. J Mater Process Technol 1998; 77:115.
- [15] Keefer D. Laser-Induced Plasmas and Applications. Radziemski LJ, Cremers DA (Ed.). New York, NY: Marcel Dekker, Inc.; 1989.
- [16] Bolshakov AP, Konov VI, Vostrikov VG, Dubrovskii VY, Kosyrev FK, Naumov VG, Shachkin LV. Quantum Electron 2008; 38:165.
- [17] Schwarz J, Meteva K, Grigat A, Schubnov A, Metev S, Vollertsen F. Diamond Relat Mater 2005; 14:305.
- [18] Varnin VP, Laptev VA, Ralchenko VG. Inorg Mater 2006; 42:S1.
- [19] Weerasinghe VM, West DRF, de Damborenea J. J Mater Process Technol 1996; 58:79.
- [20] Kato A, Tamari N. J Cryst Growth 1975; 29:55.
- [21] Kurtz SR, Gordon RG. Thin Solid Films 1986; 140:277.
- [22] Wu HZ, Chou TC, Mishra A, Anderson DR, Lampert JK, Gujrathi SC. Thin Solid Films 1990; 191:55.

INVESTIGATIONS OF LASER-SUSTAINED PLASMA AND ITS ROLE IN LASER NITRIDING OF TITANIUM

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The role of near-surface plasma during CO_2 laser nitriding of titanium is not well understood [1]. While some authors argue that plasma formation may aid the laser nitriding process [2,3-6] others ignore its role [7-16] or try to prevent its formation [17,18]. Plasma is expected to play a more important role in CO_2 laser nitriding than in nitriding with shorter wavelength lasers due to greater absorption of the laser beam within the plasma by inverse bremsstrahlung.

Some researchers attempt to suppress plasma during laser nitriding arguing that it reduces power reaching the substrate [17,18]. Others regard plasma to be beneficial arguing that near-surface plasma allows for a more efficient coupling of energy between a CO_2 laser and the surface of a metal [2,4-6]. Giren suggests that hot ions and atoms, generated by the plasma, impact the surface and activate mass-transport processes within the surface layer, which may be molten [5,6]. Others point out that UV radiation emitted from the laser plasma is absorbed more by titanium than the 10.6 µm CO_2 laser radiation [2,4]. It has been suggested that the recoil pressure, resulting from the formation of laser plasma, may also promote nitrogen incorporation and diffusion into molten titanium [2,4]. Höche and Schaaf [19] argue that plasma leads to dissociated, atomic nitrogen which more efficiently bonds with the substrate due to its higher adsorption capacity than molecular nitrogen.

The presence of plasma near a substrate surface has also been proposed as a means to reduce oxygen contamination. Liu et al. [20] and Yu et al. [21] have, respectively, demonstrated nitriding of iron and titanium by using a CO_2 laser beam and a plasma gun simultaneously in open atmosphere. Yu et al. [21] argue that, in open atmosphere, titanium reacts more readily with excited nitrogen atoms and ions than with the surrounding, unexcited, oxygen. Thus, the presence of nitrogen plasma above a titanium surface, coupled with surface heating and melting from a CO_2 laser, allows for titanium nitriding in open atmosphere without oxidation.

In this presentation, we will reveal a series of novel experiments to explore the hypotheses discussed above. We use a laser-sustained nitrogen plasma to investigate the effects of plasma during laser nitriding. (Laser-sustained plasma (LSP) is a laser plasma that can be struck and sustained indefinitely near the focus of a high-powered CO_2 laser in open atmosphere without enclosures or oxygen shielding devices.) We explore conditions where (1) titanium metal can be nitrided using a pre-struck lasersustained nitrogen plasma traversing a titanium substrate, and (2) comparable conditions where nitriding occurs without a plasma being struck at the surface. These experiments enable us to compare the resultant microstructures and explore the mechanisms of nitride formation with and without plasma. We will also explore the reactant species in the plasma and near-surface regions using CCD imaging to shed further light on the nitriding mechanisms. These experiments will provide new insights regarding the role of plasma absorption, the importance of nitrogen dissociation during the laser nitriding process and the role of plasma in transferring energy to substrate. The results will be applied to identify optimal processing regimes for the formation of near-stoichiometric titanium nitride surfaces with minimal oxidation during CO_2 laser nitriding.

Our results indicate that:

- laser-sustained plasma provides a novel tool for studying the formation of hard coatings such as nitrides and oxynitrides;
- a threshold translation speed was identified, as a function of off-focal distance;
- above the threshold speed, nitrogen-rich pre-struck plasma was located near the surface of the substrate;
- above the threshold speed, a near-stoichiometric titanium nitride surface with dendritic sub-surface structures was formed in open atmosphere;
- below the threshold speed, the surface showed a higher susceptibility to oxidation and compositional variation;
- the presence of atomic nitrogen can arise from either dissociation of molecular nitrogen in the laser-sustained plasma or at the melt surface, with subsequent incorporation into the melt trail;
- conditions were identified where the degree of titanium nitride formation within the melt was similar for dissociation taking place within the plasma (i.e. above the surface) or at the melt surface, where decomposition of nitrogen in the melt is the rate limiting step;
- energy input into and its distribution within the sample was not attenuated by the existence of a nitrogen-rich plasma; and
- there was evidence for reaction of nitrogen within the plasma with titanium vaporized from the substrate.

Figure 1 provides guidance, with respect to the off-focal distance and translation speed necessary for the formation of near-stoichiometric titanium nitride coatings. The presented results have important implications for surface processing. For the first time, we distinguish the effects under three

modes of processing: 1) processing using a pre-struck LSP, 2) processing with laser beam, without plasma formation and 3) processing with a surface-generated plasma.

Word Count: 789



Figure 1: Diagram of regions observed during CO_2 laser nitriding. Black powder was observed alongside processed trails in the striped region. Below the sold, black curve, oxide was observed within the processed sample. Below the dashed, gray curve, plasma was observed during processing. Above both the black, solid and gray, dashed curves, oxygen contamination was prevented via pre-struck LSP processing.

- Schaaf P 2000 Laser nitriding of metals Prog Mater Sci 47 1-161
- [2] Thomann A L, Sicard E, Boulmer-Leborgne C, Vivien C, Hermann J, Andreazza-Vignolle C, Andreazza P and Meneau C 1997 Surface nitriding of titanium and aluminium by laser-induced plasma Surf. Coat. Technol. 97 448-52
- [3] Akarapu R K, Dua P, Campbell A, Scott D, Nassar A, Todd J and Copley S 2008 Characterization of spectral emissions from laser irradiated titanium (Boston, MA) Mater Res Soc Symp Proc 1040 25-30
- [4] Thomann A L, Boulmer-Leborgne C, Andreazza-Vignolle C, Andreazza P, Hermann J and Blondiaux G 1996 Metal surface nitriding by laser-induced plasma J. Appl. Phys. 80 4673-84.
- [5] Giren B G 1993 Steel surface processing by a continous optical discharge plasma. Plasma Chem. Plasma Process. 13 133-40.
- [6] Giren B G 1991 Absorption of laser radiation in an optical discharge plasma in a gas mixture stream J. Phys. D, Appl. Phys. 24 1086-7.
- [7] Mordike B L 1986 Laser gas alloying NATO ASI Series, Series E: Applied Sciences. Draper C W, Mazzoldi P (San Miniato, Italy) pp 389-412

- [8] Xue L, Islam M U, Koul A K., Bibby M and Wallace W 1997 Laser gas nitriding of Ti-6Al-4V alloy part 1: Optimization of the process Adv Perform Mater 4 25-47
- [9] Nwobu A I P, Rawlings R D and West D R F 1999 Nitride formation in titanium based substrates during laser surface melting in nitrogen-argon atmospheres Acta Mater 47 631-43
- [10] Raaif M, El-Hossary F M, Negm N Z, Khalil S M, Kolitsch A, Höche D, Kaspar J, Mandl S and Schaaf P 2008 CO2 laser nitriding of titanium J. Phys. D, Appl. Phys 41 085208-1-8
- [11] Selamat M S, Baker T N and Watson L M 2001 Study of the surface layer formed by the laser processing of Ti-6Al-4V alloy in a dilute nitrogen envirnment J Mater Process Technol 113 509-15
- [12] Weerasinghe V M, West D R F and de Damborenea J 1996 Laser surface nitriding of titanium and titanium alloy J Mater Process Technol 58 79-86
- [13] Jianglong L, Qiquan L and Zhirong Z 1993 Laser gas alloying of titanium alloy with nitrogen Surf. Coat. Technol. 57 191-5
- [14] Bonss S, Brenner B and Beyer E 2001 Laser gas alloying of titanium - New possibilities for severe wear loaded components in medicine (Gothenburg, Sweden) 311-9
- [15] Mridha S and Baker T N 1994 Crack-free hard surfaces produced by laser nitriding of commercial purity titanium Mater Sci Eng A Struct Mater Prop Microstruct Process 188 229-39.
- [16] Mridha S and Baker T N 1991 Characteristic features of laser-nitrided surfaces of two titanium alloys Mater Sci Eng A Struct Mater Prop Microstruct Process A142 115-24.
- [17] Abboud J H, Fidel A F and Benyounis K Y 2008 Surface nitriding of Ti-6Al-4V alloy with a high power CO2 laser Opt Laser Technol 40 405-14.
- [18] Kloosterman A B and De Hosson J T M 1995 Microstructural characterization of laser nitrided titanium Scr. Metall. Mater 33 567-73
- [19] Höche D and Schaaf P 2011 Laser nitriding: Investigations on the model system TiN. A review Heat Mass Transfer 47 519-40
- [20] Liu J, Sun F and Yu H 2005 Nitridation of iron by the mixing technology with laser and plasma beams Appl. Surf. Sci. 252 921-28.
- [21] Yu H, Sun F and Zhang J 2009 Laser and plasma nitriding of titanium using CW-CO2 lasers in the atmosphere Curr. Appl. Phys 9 227-33.

Paper Poster

Presentation and Poster DRAFT ABSTRACT

ESM TODAY FULL-FIELD STRAIN RECOVERY IN NITI

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Shape memory alloys (SMAs) are a unique class of alloys with the ability to recover strains as large as 12% after seemingly 'plastic' deformations. This recovery is due to the martensitic phase transformation (MT), which is crystallographically reversible in SMAs [1]. The MT is a diffusionless, solid-to-solid phase transformation from the parent austenitic phase to the martensite phase. The two properties of SMAs due to which the strain is recovered are the shape memory effect and pseudoelasticity (Figure 1). In the shape memory effect, mechanically induced 'plastic' strains after unloading are by heating above a critical temperature (A to B in Figure 1). Pseudoelasticity refers to the materials ability to recover stress induced strain on mechanically unloading the specimen, and this effect occurs at a higher temperature regime than the shape memory effect (C to D in Figure 1). Nickel-titanium or nitinol (NiTi) is one of the most successful SMAs used in applications in the fields of aerospace, civil and biomedical engineering. Experimental studies have addressed the strain localization accompanying the macroscopic strain recovery due to pseudoelasticity in NiTi [2–5], but little attention has been given to the strain recovery associated with the

shape memory effect. Experimental data for localized strain in NiTi would provide better insights for its further development and use in engineering applications.

Digital image correlation (DIC) is a non-contact optical technique used to obtain full-field strain measurements [6]. In DIC, a digital camera is used to photograph the surface of a specimen. The surface of the specimen is covered with a speckle pattern (Figure 2), which is photographed before and after deformation. By comparing the pixel intensity data from deformed to the undeformed image, full-field strain fields displaying localized measurements can be obtained.

In this study, we use DIC to make localized measurements of strain during MT in NiTi. The results shows that the MT starts with the nucleation and growth of high strain bands after elastic deformation, and the seemingly permanent localized strain after unloading is completely recovered to the initial undeformed state on heating.

Word Count: 342



Figure 1: Shape memory effect and pseudoelasticity in NiTi. A to B is strain recovery due to heating (Shape Memory Effect). C to D is strain recovery due to unloading (Pseudoelasticity).



Figure 2: Speckle pattern on NiTi tensile specimen. An extensometer is attached to the specimen to record macroscopic strain.

REFERENCES

- [1] K. Otsuka and C. Wayman, *Shape Memory Materials*. Cambridge University Press, 1998.
- [2] S. Daly, G. Ravichandran, and K. Bhattacharya, "Stress-induced martensitic phase transformation in thin sheets of Nitinol," *Acta Materialia*, vol. 55, no. 10, pp. 3593-3600, Jun. 2007.
- [3] A. Schaefer and M. F.-X. Wagner, "Strain mapping at propagating interfaces in pseudoelastic NiTi," *ESOMAT 2009 8th European Symposium on Martensitic Transformations*, vol. 6031, pp. 1-6, Sep. 2009.
- [4] K. Kim and S. Daly, "Martensite Strain Memory in the Shape Memory Alloy Nickel-Titanium Under Mechanical Cycling," *Experimental Mechanics*, vol. 51, no. 4, pp. 641-652, Dec. 2010.
- [5] J. A. Shaw and S. Kyriakides, "On the Nuceleation and Propagation of Phase Transformation Fronts in a NiTi Alloy," *Acta Metallurgica*, vol. 45, no. 2, pp. 683-700, 1997.
- [6] J. Dally and W. Riley, *Experimental Stress Analysis*, 4th ed. College House, pp. 581-601.

Presentation and Poster DRAFT ABSTRACT

Light-driven tunable dual-band plasmonic absorber using liquidcrystal-coated asymmetric nanodisk array

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Abstract:

We experimentally demonstrated a light-driven reconfigurable near perfect plasmonic absorber working at dual frequencies in infrared range. By employing nanodisks with different sizes in certain arrangement, near perfect absorption of incident electromagnetic waves can be achieved for different working frequencies due to the resonance between the incident light and the nanodisk of different sizes. We showed that optically induced changes in the dielectric constant of the adjacent liquid crystal layer is an effective means to tune the absorption bands of an asymmetric gold nanodisk array. Our liquid crystal based infrared plasmonic absorber can be tuned by using visible light in real time. A tunable range of 25 nm has been confirmed by both simulation and experiment.



POSTERS

Poster

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PROGRAMMABLE CHEMICAL WAVEFORM AND SWITCHING USING ACOUSTICALLY ACTIVATED BUBBLES

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Generating chemical waveforms and switches in microfluidic devices with the flexibility to control the various signal attributes has important implications for high-throughput characterization of dynamic chemical¹ and biological processes². For example, dynamic switching or concurrent application of different chemical stimuli is valuable for studying dvnamic systems such as cell signaling pathways¹ or of chemical reactions.² cascades Although considerable progress has been achieved in spatial regulation of chemical stimuli, i.e., generation of chemical gradients¹, the temporal regulation has received little attention.

In this work we demonstrated programmable chemical waveform and switching generation using acoustically activated bubbles. Our technology has excellent control over both special and temporal regulation. The schematic diagram of our chemical waveform concept is shown in Fig. 1a. The two inlets were infused with dye and buffer solutions respectively. An air bubble was trapped at the fluidfluid interface within the horse-shoe structure (HSS). An air bubble trapped in a liquid medium acts as a focusing agent of acoustic energy in the presence of an acoustic field. Upon activation of the bubble, the microstreaming rapidly transported and interchanged the liquids in the double-ring recirculation regions. Consequently, when the bubble stops oscillating, the mixing developed by microstreaming stops and the concentrations revert to their original states. In our approach, direct translation of electrical signals into chemical waveforms enables programming all the typical features of a function generator (Fig. 2).

Fig. 3a shows the schematic diagram of our approach on demonstrating switching between two different

chemical signals, i.e. a binary logic circuit. Two HSS traps with different dimensions were positioned at the two liquid-liquid interfaces, with bubble resonant frequencies of 29.5 kHz and 14.7 kHz respectively (Fig. 3a). Cross-excitation of these bubbles at the two frequencies was negligible as determined by a microstreaming bead test (Fig. 3b). When bubble A is activated alone at f = 14.7 kHz, the red dye mixed with water, filling the region of interest with red dye (Fig. 3c and d, bottom panel). Conversely, when bubble B is activated alone at f = 29.5 kHz, the blue dye mixes with water filling the region of interest with blue dye (Fig. 3d, top panel). Switching between the red and blue dyes was achieved by programming the piezoelectric transducer to alternate between the two excitation frequencies in an oscillatory fashion, such that when bubble A is excited, bubble B remains stationary, and vice versa (Fig. 3e). One could program the transducer to achieve any desired combination of the two signals and each signal may independently access all of the functions of the waveform generator, including frequency and amplitude modulation.

In conclusion, using acoustically activated bubbles, we demonstrated automatic modulation of various signal characteristics including frequency (up to 45 Hz), amplitude, pulse width, duty cycle, and shape. We also demonstrate the applicability of the current design for dynamic switching between two distinct chemical. With its strong fucntionalities and simple design, our approach is expected to be valuable in high-throughput characterization of many dynamic chemical and biological processes.

Word Counts: 497

REFERENCES:

 Rocheleau, J. V. *et al.*, Microfluidic glucose stimulation reveals limited coordination on intracellular Ca2+ activity oscillations in pancreatic islet. *Proc. Natl Acad. Sci. USA* **101**, 12899-12903 (2004).
 T.S Shimizu, Y. Tu, and H. C Berg, "A modular gradient-sensing network for chemotaxis in Escherichia coli revealed by responses to time-varying stimuli". *Molecular system Biology*, 2010, **6:382**. doi:10.1038/msb.2010.37





Figure 1: Concept of waveform generation. (a) Schematic of the experimental setup. The piezoelectric transducer, which generates low intensity acoustic waves, is placed adjacent to the microfluidic channel on a cover glass slide. The acoustic waves drive the bubble trapped in the HSS. (b) Microstreaming on particles during bubble oscillation on the x-y plane. (c-i) The mixing of red and blue dyes by microstreaming is captured by high-speed imaging technique.

Figure 2: Generation of various chemical waveforms. The region of interest for the output waveform was chosen 500 μ m past the recirculation zone, in the bottom half of the channel, Fig 1(c). (a) Square wave. (b) Burst mode (c) Duty cycle. (d) Tunable frequency. (e) Graph of flow rate utilized in achieving amplitude modulation.(f) Sine wave.



Figure 5: Bubble based switching of blue and red food dyes. (a) Schematic of the experiment setup. The microfluidic fluidic channel contains HSSs of different. (b), Top, microstreaming visualization (red) of bubble trapped in HSS: A, while no streaming is observed in bubble trapped in HSS: B at an excitation frequency of 14.7 kHz. Bottom, microstreaming effect (blue) in bubble trapped in HSS: B at an excitation frequency of 29.5 kHz. (c) Table showing the concept of binary logic circuitry. (d) Result showing the switching mechanism between blue and red dyes. (e) Graph of experimental data for switching between red and blue food dyes in our selected ROI marked in d.

IMPROVED NANOCOMPOSITE VO_x THIN FILMS FOR MICROBOLOMETER APPLICATION

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Ion Beam Deposition (IBD) is a well-established method of growth of *better* resistivity, TCR and 1/f-noise characteristics of VO_x thin films for use in microbolometer application. Previous work on pulsed DC magnetron sputtered VO_x films has shown the films to contain voids and have a lower density. However, in this work we have explored the possibility of producing reproducing/surpassing IBD films by ion bombardment in pulsed DC magnetron sputtering. An *rf* power supply has been used to bias the substrate during reactive magnetron sputtering in a Kurt J. Lesker CMS-18 system.

The TCR - resistivity characteristics for 500Å thick films showed an inverse relationship with the applied substrate bias. The 1/f noise characteristics, as shown in Figure 1 of films with substrate bias deposited films also improved and were comparable to those of ionbeam deposited films. Further investigation by a series of deposited films showed that the 1/f noise values for films significantly thicker than 800Å showed 1/f noise properties three orders of magnitude better than films 800Å or thinner.

Grazing incidence x-ray diffraction data in Figure 2a shows an increase in crystallinity of the *fcc* VO phase and Figure 2b shows a shift of the peaks to lower the-theta values. This shift in peak position indicates either, an increase in the oxygen content in biased films, or an increased lattice strain.

However, stoichiometric analysis of the films by RBS showed a decrease in the average oxygen percentage in the films and an increase in the density of the deposited films. Thus, stress measurements were made, that confirmed that indeed films deposited with a high magnitude of substrate bias have incredibly large stress associated with them.

Microstructure analysis by TEM indicated an evolution of microstructure with the application of a substantial magnitude of substrate bias (>100V). Columnar nanotwinned grain growth is observed in substrate biased films when compared with unbiased magnetron sputtered films. It is probable that nanotwin formation may be a mechanism to compensate for the elastic energy during the thin-film growth

In conclusion, dense columnar VO_x thin films with nanotwinned microstructure (similar to IBAD films) embedded in amorphous VO_x matrix can be fabricated in pulsed DC magnetron sputtering under substrate bias conditions. These bias sputtered films show superior electrical properties when compared with unbiased magnetron sputtered and Ion-Beam deposited films (Figure 3). It was also seen that an RF substrate bias can be used to control the oxygen % in the VO_x thin films and resultant electrical and noise properties.



Figure 1: Normalized Hooge's parameter (α_H/n) versus TCR of VO_x films deposited by various methods.



Figure 2: (a) Glancing incident X-ray diffraction patterns and (b) the associated lattice parameters of VO_x films as a function of substrate bias



Figure 3: TCR vs. ρ of VO_x thin films deposited by substrate bias deposited vs. other deposition methods

DEVELOPMENT OF A LITHOGRAPHICALLY PATTERNED THREE-ELECTRODE DEVICE FOR IN VIVO AND IN VITRO EVALUATION OF CORROSION H. A. Basantani, <u>A. G. Hartsock</u>, B. A. Bimber, D. R. Cook, B. A. Shaw, M. W. Horn, E. Sikora Engineering Science and Mechanics Department, Penn State, PA-16802, USA

Biomedical implants are medical devices that are inserted into the human body. These devices, or implants, provide structure and support for ailing tissue. In most situations, an implant remains in the host's body for long periods of time; either for the remainder of the host's life (cardiac stents) or until the tissue is healed (bone plates). The age of patients needing implants has decreased over the years leaving a growing concern about the effects of permanent implants [1]. New bioabsorbable alloys are under investigation to accommodate this need for temporary implants. Currently, magnesium (Mg) and its alloys are being considered for the task since they have a very high corrosion rate [2-5]. There are also physiological benefits for this material selection including healthy bone growth [7] and a decrease in cardiovascular disease [7].

The corrosion rate of a material corresponds to its disintegration and can be measured using electrochemical techniques. This rate can be used to determine the material's lifespan in an environment. Permanent biomedical devices should have a long lifespan, or low corrosion rate, to ensure lifelong support. However, for temporary devices, high corrosion rates in a host environment are crucial.

A three-electrode device was developed for *in vitro* and *in vivo* electrochemical testing. Lithographic processing was used in the fabrication of the planar chip (Fig 1). The small design will eventually allow for *in vivo* experiments to be conducted on rat skulls. The chip was designed to fit on a male wistar's (rat) skull. Figure 4 details the stack used in the implant device on previously mentioned male wistar's skull. Lithographic processing provides precise electrode areas, which is important for crevice corrosion prevention [8]. The metal electrodes are deposited onto the silicon dioxide substrate via physical vapor deposition (PVD). PVD provides uniform and contaminate free thin films for corrosion testing. Before any animal

testing occurs, out-of-host (*in vitro*) experiments must be consistent and reliable. Sputtered titanium (Ti) devices were tested as a control reference. Titanium is a common biomaterial that is currently used in biomedical devices because of its lack deterioration in body fluid. Platinum was used as a quasi-reference electrode and counter electrode due to its stability (theoretical) in host conditions.

Using Gamry Electrochemistry Software, open circuit potentials (OCP) and polarization resistance measurements were conducted on the Ti devices. Polarization resistance values (R_p) are used in calculating the corrosion rate of a material by substituting them into Equation 1 [8], where the Tafel slopes (β_a and β_c) are 0.1 (V/Dec). Equation 2 then uses the i_{corr} rate to calculate the corrosion rate in mpy [8].

$$i_{corr} = \frac{\beta_a \times \beta_c}{2.3 \times R_p \times (\beta_a + \beta_c)} \tag{1}$$

$$CR_{mpy} = \frac{0.129 \times l_{corr} \times EW}{D} \tag{2}$$

The average corrosion rate for one of the Ti devices at 37°C Hank's Balanced Salt Solution (HBSS, simulated body fluid) was $0.7574 \text{ mpy} \pm 0.1509$ mpy. A published corrosion rate for a Ti biomaterial alloy was 0.007 mpy in 37°C deaerated HBSS [9]. The difference in these two corrosion rates could be due to the de-aeration of the solution. The quasi-reference material could also be responsible for differing corrosion rates. The sputtered Pt material used has a drifting corrosion potential. However, polarization resistance testing is about 3.5 minutes in length. The quasi reference electrode is relatively stable during the corrosion testing. Therefore, the corrosion rates found for the titanium control devices are valid. Further testing of the control devices and of the Mg alloys, will provide more corrosion rates and more confidence in the results. Word Count: 568



Figure 1. Lithographically patterned implant device along side a ruler illustrating dimensions of 9mm by 6mm



Figure 2. A zoomed side-view of a male wistar's skull showing the implant device stack and structure.

[1] B.A. Shaw, E. Sikora, S. Virtanen, "Fix, Heal, and Disappear: A New Approach to Using Metals in the Human Body," *The Electrochemical Society: Interface*, Summer, pp. 45-49, 2008

[4] F. Witte, N. Hort, C. Vogt, and et al., "Degradable biomaterials based on magnesium corrosion," *Current Opinion in Solid State and Materials Science*, vol. 12, pp. 63–72, 2008.

[5] R. Zeng, W. Dietzel, F. Witte, N. Hort, and C. Blawert, "Progress and challenge for magnesium alloys as biomaterials," *Advanced Engineering Materials*, vol. 10, no. 8, pp. B3–B14, 2008.

[6] Office of Dietary Supplements, "Magnesium: Dietary supplement fact sheet," July 2009.

[7] J. J. Vitale, "Magnesium deficiency and cardiovascular disease," *Lancet*, vol. 340, pp. 1224, 1992.

[8] D. Jones, Principles and Prevention of Corrosion. Prentice-Hall, Inc, 2 ed., 1996.

[9] I. Gurappa, "Characterization of different materials for corrosion resistance under simulated body fluid conditions," *Materials Characterization*, vol. 49, pp. 73-79, 2002

^[2] M. Staiger, A. Pietak, J. Huadmai, and G. Dias, "Magnesium and its alloys as biomaterials: A review," *Biomaterials*, vol. 27, pp. 1728–1734, 2006

^[3] F. Witte, V. Kaese, H. Haferkamp, and et al, "In vivo corrosion of four magnesium alloys and the associated bone response," *Biomaterials*, vol. 26, pp. 3557–3563, 2008.

RADIO FREQUENCY COIL DESIGN FOR MAGNETIC RESONANCE IMAGING (MRI)

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Introduction: MRI is one of the major imaging techniques in the daily clinical routine. The B0 of clinical scanners range from 0.5 to 3 T, and the B1 frequencies are therefore 21 and 128 MHz, respectively. Penn State has a 14T and 20T scanner with frequencies of 600 MHz and 850 MHz. RF resonator design becomes more challenging as the RF increases.

Methods: All simulations are conducted using CST (A 3D electromagnetic simulation software). The inherent shift of the frequency needs to be corrected with tuning capacitors which are not included in the model as of jet. In the model, the capacitance can simply be changed by extending the gap between the conductors. The two dimension magnetic field map at resonant frequency (600MHz) is plotted in Figure 1. According to the simulation, the strong magnetic field (1786A/M) if uniformly distributed inside the coil.



Fig1. (left) Solid model of the RF coil made by CST. (right) at the resonant frequency, the magnetic field inside the coil.

Discussion: There are still potential modifications to this design. The capacitors could be improved into adjustable capacitor to change the resonant frequency, and a copper foil circle instead of the two circles inductor may produce a more homogeneous magnetic field.

EFFECTS OF OXYGEN INLET POSITION AND SUBSTRATE BIAS ON PULSED DC MAGNETRON SPUTTERED VANADIUM OXIDE THIN FILMS

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Vanadium oxide thin films have been used as the main sensing materials in uncooled microbolometers for several years. Vanadium has multi valence states and vanadium oxide can transit from on phase to another very quickly under different oxygen level. The resistivity and temperature coefficient of resistivity (TCR) of vanadium oxide can also change as a function of oxygen content. It is important to control the vanadium oxide thin film composition and reach stable electronical properties (low resistivity, high TCR and low noise). In this study, vanadium oxide thin films were prepared by reactive pulsed DC magnetron sputtering system with a metallic vanadium target at room temperature. The power supply was 300 watts; total pressure was 5 mTorr; total gas flow rate was 18 SCCM and throw distance was 8 inch. Oxygen inlet position, partial ratio, and substrate bias were varied to study the effects on vanadium oxide thin film composition and properties. Oxygen was led into the process chamber from two different positions: 1 inch below the substrate and 2 inch below target. The oxygen partial pressure was varied from 5%, 7.5%, 10%, 12% and 15% and different watts substrate bias was applied.

The cathode current was monitored under different oxygen partial ratio. The cathode current and film thickness dropped quickly when oxygen flow rate increased to certain value, which indicates that the metallic target was oxidized and deposition rate decreased. Without substrate bias, the resistivity increased more gradually when oxygen inlet was near substrate and the resistivity was lower compared to those when oxygen inlet was near the target. It shows that the target oxidation mainly controls film composition when oxygen inlet is near target whereas oxidation at substrate has strong influence on film composition when oxygen inlet is near substrate. Thus, oxygen position plays an important role on the film composition and properties. However, with substrate bias, the situation is more complicated. Vanadium oxide thin film resistivity increased more gradually with increasing oxygen level with substrate bias than without substrate bias. Though increasing substrate bias decreased thin films resistivity and TCR, the TCR dropped more slowly than resistivity. Substrate bias also decreased vanadium oxide thin film thickness. It suggests that the substrate bias could be used to improve controllability of vanadium oxide thin film composition and properties. Future work would investigate the optimal throw distance and oxygen inlet position for better vanadium thin film composition controllability.



Fig. 1 Schematic of the sputtering geometry in process chamber



Fig. 2 Resistivity vs. O_2 ratio under different O_2 position log scale



Fig. 3 TCR and Resistivity drops as increasing substrate bias

REFERENCES:

[1] R.A.Wood, "Monolithic Silicon Microbolometer Arrays", *Semiconductors and Semimetals* 47, 1997, pp. 43.

(For Poster) SINGLE-STEP HOLOGRAPHIC PATTERNING OF LARGE-AREA PERIODICALLY CORRUGATED SILVER FILMS

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Surface plasmon polaritons (SPPs) are defined by the coherent oscillation of unbound electrons at a metal-dielectric interface.[1] Many applications have been demonstrated that utilize the unique properties of SPPs.[2] For example, the extreme sensitivity of SPPs to refractive index variation in surrounding materials enables sensitive, real-time, and label-free detection for applications such as environmental monitoring, food safety, and medical diagnostics.[3–6]

To excite SPPs, a promising technique is to use sub-wavelength gratings made in thin films of high-conductivity metal.[4] To date, it remains a significant challenge to fabricate sub-wavelength metal gratings over large areas at low cost.

We have developed a simple, highthroughput, and cost-effective method to fabricate one-dimensional (1D) and twodimensional (2D) periodically corrugated silver films over centimeter scale areas. This fabrication uses a single-step of holographic pattering with laser intensities as low as 88.8 mW/cm^2 to create gratings with periodicities of approximately 570 nm. Surface plasmon polaritons (SPPs) can be excited on the gratings' surfaces and the samples show a high sensitivity of 555.28 nm/RIU (refractive index unit) to changes in the surrounding refractive index, which makers them promising for chemical/biological sensing applications.



Figure 1: Schematics of the experiment setup for fabrication of (a) 1D and (b) 2D periodically corrugated silver films. The incident laser beam from the bottom of the prism was split at the prism surface to form interference patterns on the reservoir containing Ag(NH3)2+ and glucose mixture. The interference patterns were demonstrated in the glass slides. Silver accumulated faster at where the light intensity was high, resulting in a silver film with surface profile following the interference pattern. (c) and (d) are the AFM images showing the surface profile of the 1D and 2D periodically corrugated silver film, respectively. Both (e) 1D and (f) 2D periodically corrugated silver films showed diffraction colors under white light illumination.



Figure 2: (a) Experimental transmission intensity of the 1D periodically corrugated silver film as a function of wavelength and incident angle, with theoretical calculations for the SPs at silver/air interface (dashed line) and silver/glass interface (dashed dotted line). (b) At normal incidence, the transmission spectra of the 1D periodically corrugate silver film immersed into air, DI water, and 3.42 M CaCl2 solution.

REFERENCES:

- T. W. Ebbesen, H. J. Lezec, H. F. Ghaemi, T. Thio, and P. A. Wolff, "Extraordinary optical transmission through subwavelength hole arrays," Nature, vol. 391, p. 667, 1998.
- [2] X. Luo, J. Ma, and T. Ishihara, "Plasmonrelated optical properties of unpenetrated metallic periodic structures," Opt. Mater., vol. 29, no. 2-3, p. 211, Nov. 2006.
- [3] J. Homola, "Surface plasmon resonance sensors for detection of chemical and biological species.," Chem. Rev., vol. 108, no. 2, p. 462, Feb. 2008.
- J. Homola, S. S. Yee, and G. Gauglitz,
 "Surface plasmon resonance sensors: review," Sensor Actual. B-Chemical, vol. 54, no. 1-2, p. 3, Jan. 1999.
- [5] J. Yao, A.-P. Le, S. K. Gray, J. S. Moore, J. A. Rogers, and R. G. Nuzzo, "Functional nanostructured plasmonic materials.," Adv. Mater., vol. 22, no. 10, pp. 1102-1110, Mar. 2010.
- [6] J. C. Love, L. A. Estroff, J. K. Kriebel, R. G. Nuzzo, and G. M. Whitesides, "Selfassembled monolayers of thiolates on metals as a form of nanotechnology.," Chem. Rev., vol. 105, no. 4, pp. 1103-1169, Apr. 2005.

INTERGRATED RT-PCR-ROSGENE STRIP DEVICE FOR INFLUENZA H1N1 VIRUS DETECTION

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Abstract

In this study, we developed a RT-PCR microdevice which is integrated with a ROSGENE strip for colorimetric detection of Influenza H1N1 virus. The **RT-PCR** microdevice consists of а pneumatic micropump for sample loading, a 1-µL RT-PCR chamber for target gene amplification, and a resistance temperature detector for temperature control as shown in Figure 1. A ROSGENE strip was connected at the outlet reservoir, and the eluted amplicons were detected as a line on the strip with naked eyes. The off-chip RT-PCR amplicons of the influenza H1N1 virus were analyzed on the ROSGENE strip in comparison with the agarose gel electrophoresis as shown in Figure 2. The test line (T) along with the control line (C) appeared, and the detection sensitivity was better than that of the agarose gel electrophoresis. The test line on the ROSGENE strip could be observed with the RNA template of 1 pg. The on-chip RT-PCR-ROSGENE strip detection was performed by injecting the RT-PCR cocktail into the chamber through the waste outlet

reservoir with the micro-pump actuation. During a PCR thermal cycling for 2 h, all the microvalves were closed to isolate the PCR chamber, and then the RT-PCR product was exported to the attached ROSGENE strip through the outlet reservior. After dropping 60 μ l of eluant buffer at the end of the strip, the test line was detected as a H1N1 virus indicator, while the negative experiment only revealed a control line as shown in Figure 3.

Poster



Figure 1. The scheme of the integrated RT-PCR-ROSGENE strip microdevice.



Figure 2. The comparison of the detection sensitivity between the agaros gel electrophoresis (A) and the ROSGENE strip (B).



Figure 3. The detection of the H1N1 influenza virus on the integrated microdevice (A) with a negative control experiment (B)

Poster DRAFT ABSTRACT

Pt AND Cu STFS FOR SOLAR CELL ELECTRODES AND CO₂ TO METHANE CONVERSION

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Sculptured thin films (STFs) can be engineered to yield specific structure in thin films, using oblique angle vapor deposition as in Fig. 1. Physically deposited electrodes show porous and columnar structures due to self-shadowing of incident vapor flux. The porosity and structural shape of the films are controllable, depending on the angle of the incident vapor flux, χ_v [1]. These controlled porosity and structural shape of enhancing their performance. In this study, Pt and Cu STFs were applied to Dye-sensitized solar cells (DSSCs) and to the film of CO₂ conversion to methane.

DSSCs have been studied extensively as a possible low-cost photovoltaic alternative. The counter electrode is one of important part of DSSCs relating to the reduction of I_3^- near its surface [2]. An enhanced surface area can result in more vigorous reactivity of the electrolyte at the counter electrode and thus can contribute to improvement of DSSCs. The structure of conventional counter electrodes is typically planar and the platinum catalyst is usually just chemically applied to either a transparent conductor or a thin layer of planar platinum. In this study, platinum films were sculptured by electron beam evaporation with a 5° vapor incidence angle onto a Pt planar electrode as counter electrodes for

DSSCs. In addition, the counter electrodes treated with chloroplatinic acid (H₂PtCl₆ in isopropyl alcohol) were prepared to investigate its effects on the films. In electrochemical impedance spectroscopy (EIS) with symmetrical cells fabricated with these six different kinds of electrodes, Pt STFs showed five times less charge transfer resistance than that of Pt planar electrode (0.578 $[\Omega^* \text{cm}^2] \rightarrow 0.121 [\Omega^* \text{cm}^2]$). Pt STFs with the treatment of H₂PtCl₆ solution showed 0.04025 [Ω^* cm²]. As shown in Fig. 2, the DSSCs with Pt STFs led to 15 [%] and 18 [%] increase in current density and efficiency respectively in comparison with Pt planar counter electrodes. The efficiency was 5.1 [%] under AM 1.5 illumination. With acid treatment, the efficiency of DSSCs reached to 5.63 [%].

Another application by using STFs is the film for CO_2 conversion to methane. In this film, cocatalyst such as Pt and Cu plays an important role in improving the performance of TiO₂ catalyst for CO₂ to hydrocarbon fuel such as methane [3]. While conventional method for depositing cocatalysts is deposited in planar, the cocatalysts are capable of being sculptured to obtain much amount of catalytic materials without preventing the flow of gas species in the chamber and also, the structure of STFs

themselves have an advantages in the aspect of high surface area that is essential for catalytic effect. For experiment, Pt and Cu STFs were grown on porous TiO₂ films structure also with the vapor flux at 5° incidence angle as shown in Fig. 3. TiO₂ film incorporating Pt and Cu STFs produced more than two times and five times methane than that of bare TiO₂ films respectively and their efficiency were 3.03ppm cm⁻² h⁻¹ and 1.25ppm cm⁻² h⁻¹ in each reaction as shown in Fig. 4.



Fig. 1 Fabrication of sculptured thin films by electron beam evaporation



Fig. 2 J-V characterisitics of DSSCs



Fig. 3 Structure of the film for CO₂ conversion



Fig. 4 Methane production rate of TiO_2/Cu , TiO_2/Pt and TiO_2

Reference

 S. Pursel, M. Horn, "Prospects for nanowire sculptured-thin-film devices," *J. Vac. Sci. Technol. B*, vol. 25, pp. 2611-2615, 2007

[2] B, O'regan, M. Grätzel, "A low cost, high efficiency solar cell based on dye-sensitized colloidal TiO₂ films," *Nature*, vol. 353, pp. 737-740, 1991)

[3] S. Roy, O. Varghese, M. Paulose, C. Grimes, "Toward Solar Fuels: Photocatalytic Conversion of Carbon Dioxide to Hydrocarbons," *ACS Nano*, vol.4, pp. 1259-1278, 2010