Electrospray from an Ionic Liquid Ferrofluid utilizing the Rosensweig Instability

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Abstract: A new type of electrospray technology that could be used for space propulsion was developed at Michigan Technological University. This thruster utilized an ionic liquid ferrofluid that was synthesized by suspending magnetic nanoparticles in an ionic liquid carrier solution so that the resulting fluid is superparamagnetic. The magnetic properties of the fluid were exploited to create self-assembling static arrays of surface peaks which were then amplified with an applied electric field until ion current was emitted from the array. The current and voltage profile of the emitting array was measured and its ability to self-heal after a damaging event was observed.

I. Introduction

Typical electrospray thruster concepts utilize linear "blade like" emitters, 2-D arrays of tips, or 2-D arrays of micro-capillaries to generate large electric fields and ion/droplet emission from an electrostatic Taylor cone. Linear arrays are manufactured from thin sheets of metal, such as porous tungsten, and sharp peaks are etched into the surface. The arrays are aligned with a slit in the extraction electrode and the propellant (ionic liquid) is passively pumped through the porous tungsten.[1, 2] This type of electrospray emitter can have good packing density along the length of the array, but leaves a sizeable gap between consecutive arrays. Legge and Lozano were able to demonstrate 2 tips per mm packing density along the length of the array.[2] A similar strategy is the tungsten crown emitter, which is a porous tungsten structure shaped like a king's crown with a circular array of 28 emitters.[3-5] The crown emitter used indium as its propellant.

Two-dimensional arrays are typically etched from silicon with regularly spaced discrete needles.[6] For this type of arrangement, the propellant is typically externally applied to the surface of the silicon where capillary flow causes a uniform layer of propellant to coat the substrate and emitter tips. In order for the propellant to easily wet to the silicon surface, a surface treatment is usually necessary, such as making black silicon.[7] These arrays are then assembled with an extraction electrode and alignment/retention mechanisms.[8]

The third type of electrospray thruster is the capillary thruster. These types of thrusters generally contain the fluid in a hollow tube or needle and the electric field is created at the exit of the capillary.[9] One such device is the capillary array of 19 emitters built by Krpoun and Shea.[10] In this device each of the capillaries are etched out of silicon and then filled with silica spheres to provide a more uniform hydraulic resistance within each emitter. A micromanufactured extraction electrode was placed and aligned using ruby balls for alignment and electrical isolation. Another version of a capillary thruster was developed by Busek.[11] Each of Busek's thruster heads contained an array of 9 individually manufactured emitters assembled into an array. A third type of thruster array which does not cleanly fit into any of these groups is the porous tungsten and porous nickel planar arrays.[12-15] Regularly spaced peaks are etched into porous tungsten and nickel. The peaks provide the electric field enhancement so that Taylor cones form on the apex. The porous nature of the substrate causes liquid propellant to passively flow to the peaks via capillary pressure. They are micromanufactured arrays similar to the externally wetted

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silicon devices, however they behave similarly to capillary electrospray thrusters due to the mechanism of propellant delivery.

All electrospray thruster concepts to-date share a common feature: microfabricated solid structures are used as "scaffolding" to position a layer of fluid at the tip of a solid electrode. The scaffolding, either hollow capillaries or solid needles, must have a solid tip with radius no bigger than 10s of microns. Such sharp electrodes are necessary in order to create a sufficiently large electric field (~10^7 V/m) to initiate a Taylor cone instability in the liquid at the apex. The scaffolding is crucial, since classical liquids do not want to form stable sharp tips on their own unless they are shaped by an underlying solid. Electrospray thrusters thus must rely on micro-fabrication techniques, such as silicon MEMS or electrochemical etching processes, that are capable of controlling surface features at the micron level.

Recently a new class of fluid was synthesized that may enable electrospray emission without the use of solid structures to shape the fluid into a sharp tip. The fluid is created by dispersing magnetic nanoparticles within an ionic liquid and is referred to as an ionic liquid ferrofluid (ILFF). The resulting fluid retains its low vapor pressure, high conductivity, and low viscosity, however the fluid is also superparamagnetic. ILFFs can be strained by magnetic fields as well as electric fields and the interaction between these strains and the liquid surface tension produces interesting geometries and instabilities. In particular, an ILFF that is subject to a magnetic field from a permanent magnet will display a static arrangement of fluid peaks known as a Rosensweig instability (see Figure 1). Stable ILFFs that demonstrate the Rosensweig instability are a relatively new creation, first created in 2011.[16-18] Because ILFFs are electrically conductive as well as superparamagnetic, application of an electric field to the Rosensweig peaks will introduce an additional surface stress that will further amplify the peak height and, if strong enough, will eventually result in electrospray from the fluid surface without any microfabricated solid structures.

The goal of work reported here was to explore the feasibility of inducing ion emission from the peaks of a Rosensweig instability formed in an ionic liquid ferrofluid. This paper begins with a brief discussion of basic ferrofluid dynamics followed by a description of ionic liquid ferrofluids including a history of ILFF development activity. Experiments are then described wherein a concentric pool of ILFF is subject to the field from a permanent magnet in addition to an electrostatic extractor grid designed to induce electrospray.

Figure 1: Example of a Rosensweig instability in a ferrofluid exposed to the field from a permanent magnet.

II. Ferrofluid

Ferrofluids are unique liquids that do not occur in nature, but have existed since their first laboratory creation in the 1960's. Ferrofluids differ from magnetorheological fluids (MR fluids) by the size of the suspended particles: ferrofluids have particles on the order of 10nm, while particles in MR fluids can be many microns. An excellent description of ferrofluid dynamics can be found in the books by Rosensweig[19] and Odenbach.[20] Ferrofluids are formed when nanometer-sized ferromagnetic particles are permanently suspended within a carrier liquid. The particles are small enough that Brownian motion keeps them from settling out of the liquid due to gravity or magnetic forces. The particles are also small enough (~10nm) so that they possess a single magnetic domain, thus the resulting colloidal fluid is superparamagnetic. The magnetic particles are often coated with a surfactant to prevent agglomeration and sedimentation, although this isn't always necessary. Common carrier liquids are mineral oils, kerosene, and water. A newer carrier liquid recently developed was ionic liquid, which is described in Section III.

The motion and behavior of a ferrofluid is described by the field of ferrohydrodynamics, which couples the Navier-Stokes equations with the Maxwell magnetostatic relations. The fluids have rich and complex non-linear motion, since they both respond to applied magnetic fields while also altering the magnetic field because of their superparamagnetic behavior. The most well known and dramatic response of a ferrofluid is the so-called Rosensweig instability,[21, 22] which is the subject of this paper. Numerous fascinating demonstrations of this instability can be seen in classrooms and home laboratories by searching for 'ferrofluids' on YouTube. The Rosensweig instability results when a perturbation in the surface of the ferrofluid causes a bunching of the magnetic field lines as depicted in the left most image of Figure 2. The bunching of the field lines, which represents a spatial gradient in the field strength, attracts ferrofluid to the strong-field region at the tips of the perturbation. As a result, the height of the fluid perturbation will increase and the field lines will be more dramatically pinched such that the field at the tip will be increased even more, attracting more fluid to the tip ad infinitum. The instability is arrested by fluid surface tension, which seeks to minimize free surface area. When a pool of ferrofluid is subject to a moderate external magnetic field the resulting surface instability can be thought of (with extreme care) as the magnetic analog of the electrostatic Taylor cone. An image of a static Rosensweig instability is shown in the rightmost image of Figure 2. From an energy standpoint the Rosensweig instability represents a balance between the magnetic interaction energy of the fluid which is reduced by the peak geometry, and the surface tension energy, which is increased by the non-planar surface. The peaks themselves can display complicated geometry as shown Figure 3, where a secondary magnetic instability is seen on the apex of each primary tip. Depending upon the depth of the fluid pool Rosensweig instabilities can create discrete peaks having no fluid between them, as in Figure 3, or as a singular continuous fluid with spikes on the surface as in Figure 2.

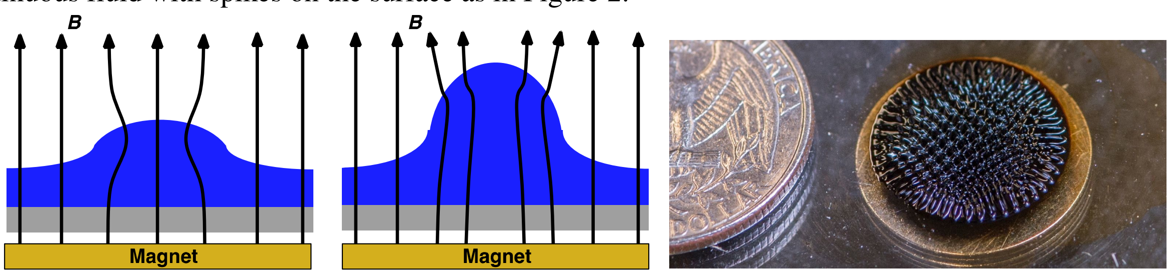

Figure 2: Left: a perturbation in the surface of the ferrofluid causes a local bunching of magnetic field lines. Center: ferrofluid attracted to the strong-field region, growing the tip height and increasing field gradient. Right: A puddle of ferrofluid is shown on a small permanent magnet (note U.S. quarter in image). The static pattern of sharp fluid spikes in the liquid surface is known as a Rosensweig instability.

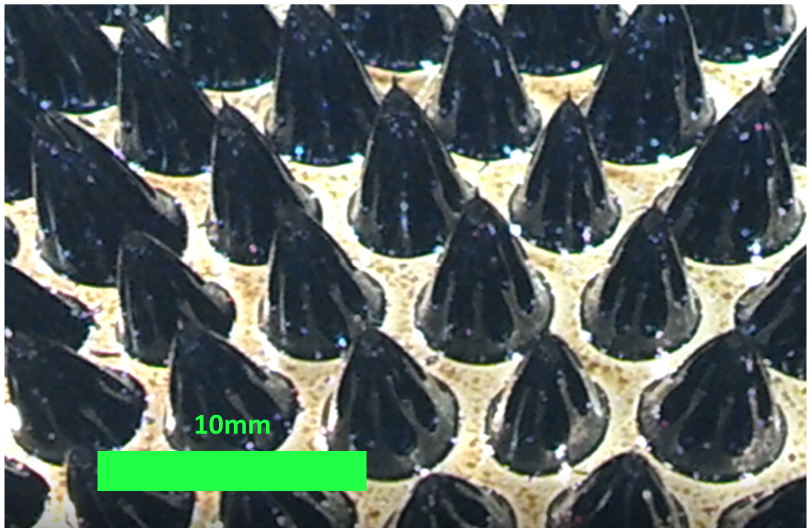

Figure 3: Close-up image of Rosensweig instability in a ferrofluid showing discrete peaks separated by regions of no fluid. Also note secondary instability peaks forming on the apex of each primary peak.

The onset and spacing of the Rosensweig instability is governed by the dispersion relation shown in Equation (1).[23] When the applied magnetic field is strong enough to create the Rosensweig instability, and the external field is uniform, such as inside a Helmholtz configuration, the spacing between tips is governed by the capillary length of the fluid, as given in Equation (2).[24] However, when the external magnetic field is non uniform, such as in the case of a permanent magnet placed behind the ferrofluid, the internal force of the magnetic field gradient can be stronger than the gravity force. In this case the tip-to-tip spacing between the peaks is no longer a function of the capillary length, but instead a function of the strength and gradient of the applied magnetic field and fluid properties, such as given in Equation (3).[25]

$$\left(1-\frac{i\omega}{2\nu^2}\right)^2+\frac{1}{4\rho\nu^2q^4}\left[\rho g q+\sigma q^3-\frac{(\mu_r-1)^2}{(\mu_r+1)\mu_0\mu_r}B^2q^2\right]=\sqrt{1-\frac{i\omega}{\nu q^2}} \quad \textbf{(1)}$$

$$\lambda_{critical}=\sqrt{\frac{\sigma}{\rho g}} \quad \textbf{(2)}$$

$$\lambda=\sqrt{\frac{\sigma}{B\nabla H}} \quad \textbf{(3)}$$

III. Ionic Liquid Ferrofluid

In 2011 Jain and Hawkett[17] successfully synthesized the first ferrofluid based on an ionic liquid carrier shortly followed by Huang and Wang in 2012[18] (note that other groups including Oliveira in 2009[26] and Rodriquez-Arco in 2011[27] have also pursued ionic liquid ferrofluids, but were unable to create stable suspensions that demonstrated the hallmark Rosensweig instability). With the addition of magnetic nanoparticles the ionic liquid still retains the characteristic properties that ionic liquids are known for, such as the low vapor pressure, liquid at room temperature, and electrical conductivity.

Jain et al were able to create a number of stable ILFFs.[17] The first two ILFFs they were able to create were using bare maghemite nanoparticles (Fe_2O_3 with no surfactants) with EMIM-Ac (1-ethyl-3-methylimidazolium acetate) and EMIM-SCN (1-ethyl-3-methylimidazolium thyocyanate). By adding a surfactant of acrylic acid-b-acrylamide copolymer to the magnetic nanoparticles, they were able to create a stable ILFF in EAN (Ethylammonium nitrate). All three of the ILFFs described were stable for at least months (they are still stable at the time of this writing) and exhibited the Rosensweig instability when a magnetic field was applied.

This paper reports on a new discovery: if an electric field is applied to a Rosensweig instability in the surface of an ILFF, the electric surface stress on the ILFF amplifies the height of the peaks and sharpens the tips until they begin to emit ions. The size and spacing of the peaks in the Rosensweig instability pattern can be modified by changing the magnetic field or container geometry. Figure 4 shows an example where tip size and spacing were controlled. In these images a series of concentric trenches were milled into acrylic, and ferrofluid was placed into the trenches with a dropper. Small permanent magnets were placed behind the acrylic. The pattern of peaks that form in the constrained trenches of Figure 4 are much different than the pattern formed in the unconstrained liquid pool shown in Figure 2. By using container geometry, peaks can be self arranged in a predictable pattern so that an extraction electrode can be sized and properly located above each ring. Using the confined geometry, the tip-to-tip spacing and the size of each tip in the trench can be modified based on the external magnetic field. In Figure 4, the applied magnetic field was varied between the left and right image, resulting in change in the instability mode.

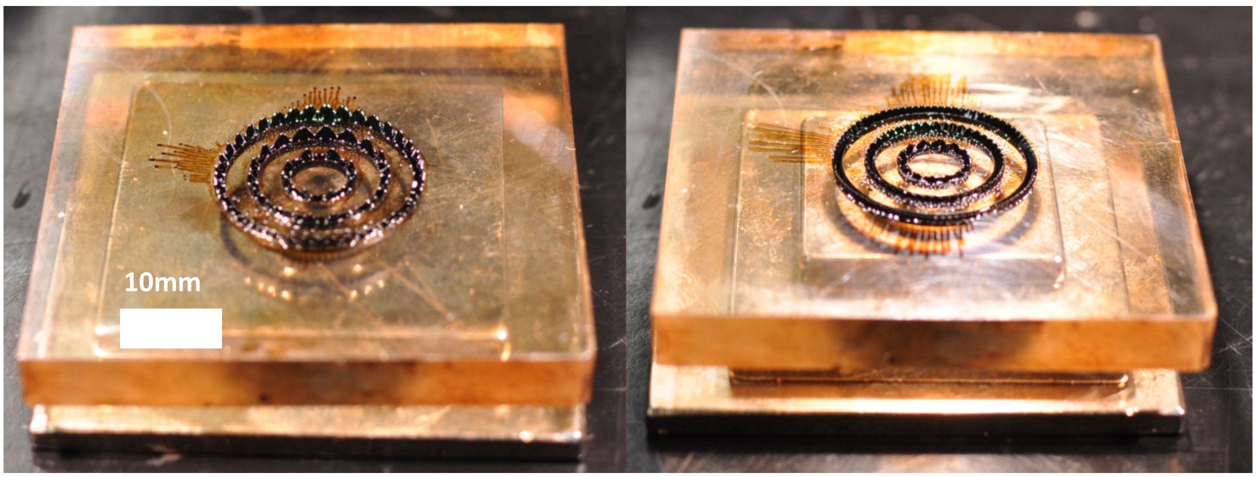

Figure 4: Variation of peak size and spacing by changing the magnetic field

IV. Experiment Design

The ILFF used in this work was developed by Brian Hawkett and Nirmesh Jain at the University of Sydney working in collaboration with Michigan Technological University. The carrier fluid of the ILFF was Ethylammonium Nitrate (EAN), with magnetic particles dispersed in the fluid with a polymer coating to prevent nanoparticles from agglomerating. The resulting ILFF was used at room temperature, however the viscosity of the fluid was observed to be higher than pure EAN. The ILFF was placed in a 2-mm-wide, 2-mm-deep, 8-mm-diameter circular trench cut into an aluminum block that provides electrical conductivity to the fluid, as can be viewed in Figure 6. An extraction electrode was placed 4.6 mm above the aluminum block containing the ILFF. The extraction electrode had a circular pattern milled into it allowing the extracted ions to pass through and be measured on a collection plate. In all of the tests, the extraction electrode was grounded, with the aluminum block holding the fluid biased to either positive or negative voltages using a Glassman Series FX or FC power supply. The collection plate was glass coated in indium tin oxide (ITO) and was electrically connected to a Keithly 2410 sourcemeter to measure the intercepted spray current. This glass was placed 1.1 mm above the extraction electrode. An illustration of the cross-section of the experimental is depicted in Figure 5.

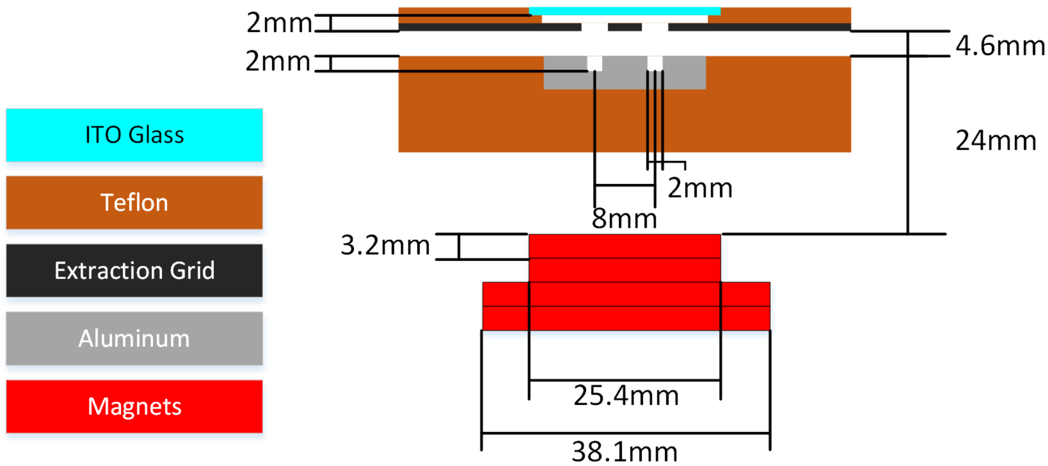

Figure 5: Illustration of cross-section of experimental setup

To induce the Rosensweig instability in the ILFF, a series of four magnets were placed 16.5 mm below the bottom of the ILFF. These magnets in order of being closest to farthest from the fluid were two 1"x1" followed by two 1.5"x1.5" and all were N52 grade, and 1/8" thick. These magnets provided a normal field of approximately 500 Gauss on the centerline at the surface of the fluid. The experimental configuration can be viewed in Figure 6.

The test apparatus was placed in an ultrahigh vacuum facility evacuated using an ion pump. The base pressure before testing was 10^{-8} Torr, but while testing, the pressure ranged between 10^{-7} and 10^{-5} Torr.

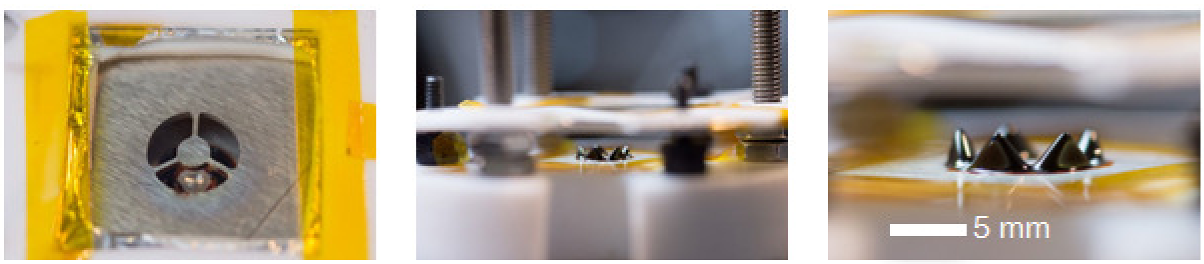

Figure 6: Experimental Setup. Left: Image taken top down, in view are the extraction electrode and ITO glass and the peaks of the ILFF. Middle: side view of extraction electrode and tips. Right: Close up image of the tips.

V. Results

When subject to the field of the permanent magnets the ILFF displayed a deformed surface of five Rosensweig peaks. An electric field was then applied by biasing the extraction electrode in an attempt to amplify the peaks and induce spray from the resulting magneto-electro-static instability. As the applied electric field was increased, the tips grew in height and became sharper (center two frames of Figure 7). When the extraction voltage reached 3700 V, the apex of each instability demonstrated an abrupt transition to a very sharp structure and began emitting ion current; this can be seen in the right most frame of Figure 7. The application of the electric field did not change the tip-to-tip spacing, nor the number of peaks, but only changed the height of the peakand their profile/curvature.

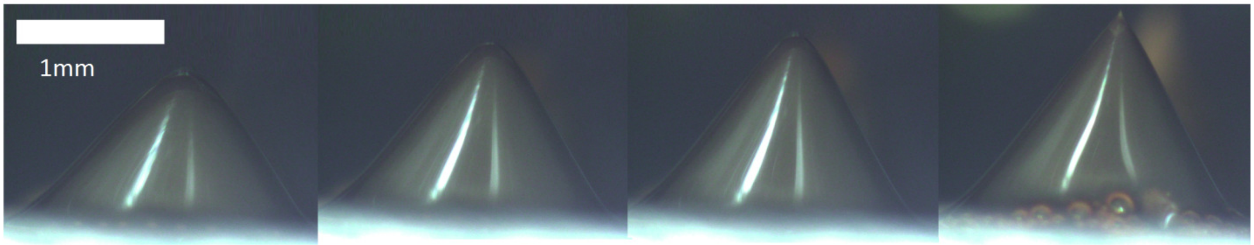

Figure 7: Images of an ILFF peak with progressive voltage being applied. From left to right: 0 V, 3000 V, 3600 V, 3700 V. Emission began at 3700 V.

A representative I-V curve of the emission can be viewed in Figure 8. The onset was at -3250 V and 1.6 μA, with a peak voltage and current of -3650 V and 14.24 μA. The turn-off voltage was 300 V lower than onset. This is partly due to the ILFF being rather viscous and partially because when the voltage is being decreased in magnitude, the peak is already formed, and the field enhancement already exists allowing it to emit at lower applied voltages. Due to variations in distance between an individual tip and the extraction grid, not all of the tips begin emitting at the same extraction voltage. Because of this, as the extraction voltage was increased, the number of existing peaks that were emitting increased. Between -3525 V and -3600 V the slope of the curve became much steeper because the voltage was high enough to cause one of the peaks to transition from a blunt, non-emitting peak, to a sharp tip emitting ions. On some of the tests, array currents up to 50 μA were measured.

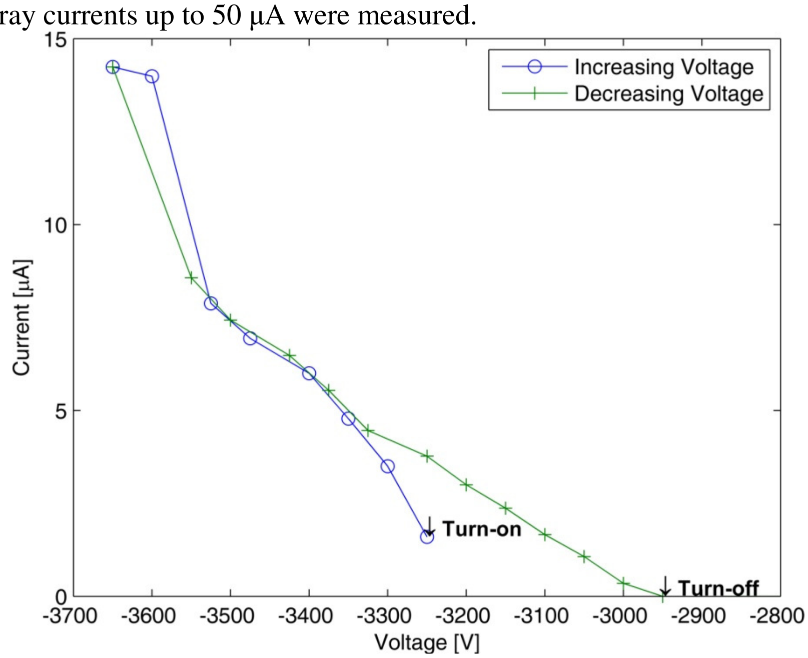

Figure 8: I-V curve of ILFF Emitting

Figure 9 shows the ITO glass current collector post test. The dark locations on the glass are the ILFF accumulating on the glass surface. There are at least 4 distinct regions on the glass where fluid was deposited suggesting at least four tips were emitting. The IL EAN itself is a clear liquid, but when doped with the magnetic nanoparticles, the ILFF is a dark brown color. Since the residue left on the extraction electrode and the ITO glass is colored brown, it is thought that at least some of the magnetic nanoparticles are emitted along with the ions.

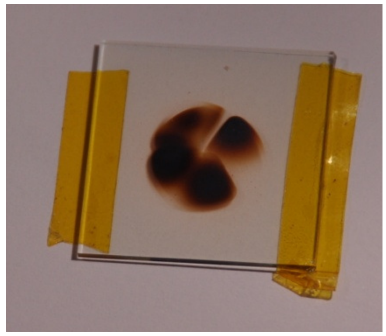

Figure 9: Current collection plate (ITO glass) showing spray from multiple emission sites

It was noted during testing that gaseous bubbles appeared in the ILFF near the base of the peaks as soon as ion emission started. It is thought that the bubbles are formed due to an electrochemical reaction between the ferrofluid and the aluminum reservoir. Occasionally the bubbles became large enough that they deformed the emitter peak and temporarily interrupted emission. However, once the bubble was "passed" by the system a new emission peak formed immediately and emission was restored. The series of images in Figure 10 show the progression of an emitting tip being damaged and then reforming and emitting again. The figure series occurred over the span of two minutes. In the left of Figure 10, the peak is emitting, but a number of bubbles can be noted at the base of the peak. When the large bubble formed, the peak nearest to the bubble became blunt and stopped emitting. About a minute later, the bubble "burst", and the tip reformed and began emitting again. This self-healing behavior was noticed in additional testing, such as when a tip arcs to the extraction electrode. Within a minute, the tip reforms and begins to emit again.

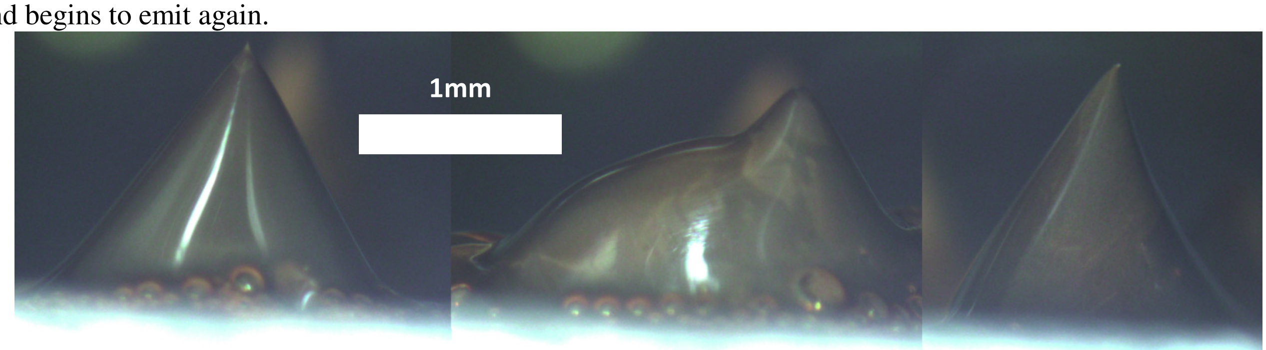

Figure 10: Left: Tip Emitting before the bubble formed. Middle: Bubble formed and tip damaged (no longer emitting). Right: Tip reformed and emitting.

Another interesting behavior that was observed was a spontaneous and unexplained bifurcation of the emission tip into multiple emission sites such as shown in Figure 11. This particular tip was observed to transition from one, to two, to three individual emission sites at the apex of the Rosensweig peak. Moreover, the appearance of multiple emission sites was accompanied by a break in the peak symmetry, which can be seen as a curved or leaning orientation. After a number of minutes and without change to the applied electric potential this peak morphed again from having three emission sites, back down to only a single emission site. The reason for the change in the number of emission sites is currently unknown, but is likely due to the strong non-linear behavior of the coupled magneto-electrostatic geometry.

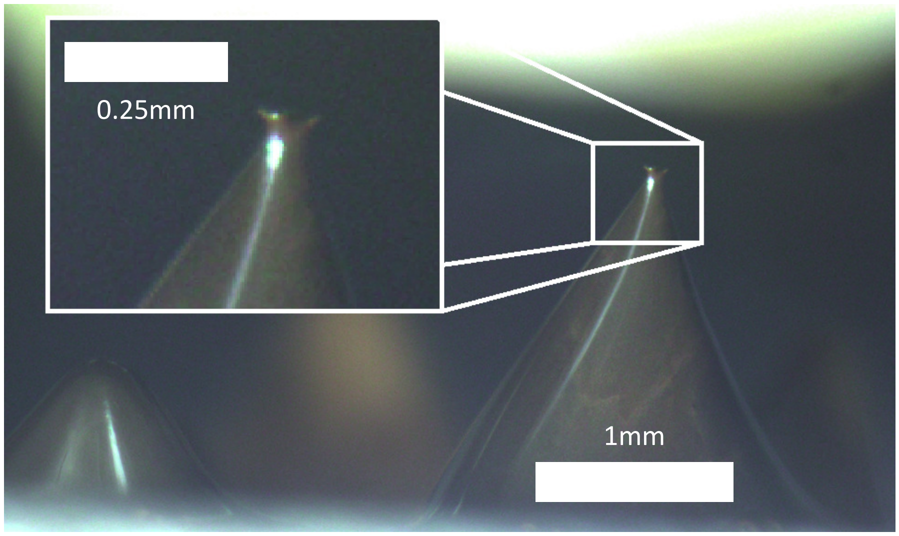

Figure 11: ILFF showing 3 separate emission sites from tip. Insert: close-up of tip showing three emission sites

VI. Conclusion

A new type of electrospray ion emission source was developed at Michigan Technological University. It utilized an ionic liquid ferrofluid and the Rosensweig instability to create a self-assembling array of liquid peaks without the need of a support structure such as a needle or capillary. The application of an electric field to the ILFF distorted the peaks to form sharp tips which led to the emission of ions from the tip. The I-V characteristic of the array was measured, along with visual evidence of multiple emission sites. Finally, during testing, it was observed that any given emitter tip could become damaged and/or destroyed, and then that same emitter tip would repair itself over the course of a few minutes and begin emitting again.

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