Characterization of an Ionic Liquid Ferrofluid Electrospray Emission Pattern

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The angular distribution of the electrospray emission plume was measured for EMIM-NTf2 based ionic liquid ferrofluid. Measurements were obtained using a tungsten collector plate which was rotated over a single ferrofluid emission source through the use of a stepper motor. Current density vs. angle was recorded for two different magnetic fields while emitting under three bias voltages. The current density was found to be rather constant within 40 degrees of the beam axis, decaying abruptly to zero for larger angles. While higher magnetic fields seemed to produce more narrow plumes, the limited data set presented here was not sufficient to allow definite correlation.

I.   Introduction

ELECTROSPRAY occurs when the meniscus of an electrically conductive or polar liquid is subjected to a large electric field. The electrostatic stress counteracts surface tension, resulting in the fluid deforming to form a sharp cone, referred to as a Taylor cone. Once a threshold field is achieved, a jet of charged particles is emitted from the tip at a high velocity. Electrospray emission can be obtained using a variety of fluids, including ionic liquids and liquid metals. Of these fluids, ionic liquids have been particularly interesting for space propulsion. Since they are composed entirely of ions, they exhibit near zero vapor pressure.

In 2013, a new technique to produce electrospray was demonstrated within the Ion Space Propulsion at Michigan Technological University. This method utilizes a fluid known as an ionic liquid ferrofluid (ILFF) as the sprayed liquid. Unlike conventional ionic liquids, ILFFs contain nanoscale ferromagnetic particles that form a colloidal mixture that is superparamagnetic and electrically conductive. In the presence of a magnetic field, a static arrangement of self-assembling fluid peaks can form, which when stressed by an electric field, can emit ions.

This method of electrospray is achieved through a combined magneto-electrostatic instability and is coined the Rosensweig-Taylor instability. This combination of magnetic with electric stress on the fluid surface makes ILFF fundamentally different than previous forms of electrospray studied to date.

Ferrofluids are superparamagnetic liquids. These fluids are composed of nanoscale ferromagnetic particles coated with a surfactant which are suspended in a carrier liquid, typically water or carbon-based solvents. Brownian motion prevents the particles from clumping or settling, even in the presence of strong magnetic fields. When these fluids are subjected to a magnetic field, a series of valleys and peaks form, which is referred to as either the normal-field instability, or the Rosensweig instability. The shapes of these peaks are dependent on fluid properties of the ferrofluid and the nature of the applied magnetic field. For conventional methods of electrospray, a supporting structure is required to achieve emission, typically consisting of either externally wetted or hollow capillary needles. These structures serve to both concentrate the electric field at the tip and transport fluid to the emission site. A feature of ILFF electrospray is that the ferrofluid can serve as both the propellant and support structure.

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Although ferrofluids have existed since the 1960's, vacuum-based applications have been hindered since typical carrier fluids will evaporate. In 2011, the first stable ILFF was developed by Jain Zhan, and Hawkett [1]. Since, stable ILFFs have been synthesized by various researchers [2-4]. When magnetic nanoparticles are suspended within an ionic liquid, the colloid maintains the low vapor pressure, high conductivity, and low viscosity of the parent ionic liquid, as well as becoming superparamagnetic. The fluid maintains its ability to be stressed by electric fields while gaining the ability to be stressed by a magnetic field.

In 2012, Meyer and King were the first to demonstrate that electrospray emission can be achieved using a ferrofluid with Ethylammonium Nitrate (EAN) as the carrier fluid [5]. Emission has since been demonstrated using 1-Ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide (EMIM-NTf2) based ferrofluid [6]. These initial studies were limited to demonstrating that emission can be achieved and establishing initial emission current versus extraction voltage relations. One area that was identified for further characterization is the effect of magnetic and electric field strength on the angular distribution of the emission current, here after referred to as the beam divergence.

Beam divergence results when charged liquid droplets and/or ions downstream from the emission point are dispersed by the nearly spherical Taylor electric field at the tip, creating an off-axis component in the droplet velocity, reducing the specific impulse and efficiency of the thruster. The specific impulse, I_{sp} , is defined as:

$$I_{sp} = \frac{v_e}{g_0} \tag{1}$$

where v_e is the average exhaust speed parallel to the desired thrust vector and g_0 is the gravitational acceleration at the Earth's surface. An angular component of the exhaust plume will decrease the effective average value of v_e and also will put power into non-thrust-producing off-axis directions reducing system efficiency. Additionally, an understanding of the beam divergence is critical for properly sizing the aperture of the extraction electrode to prevent interference with the emission stream.

Chiu [7] and Gamero-Castaño [8, 9] both experimentally investigated the emission divergence of an electrospray beam by rotating the source with respect to a fixed collector plate. Morris [10] utilized a 3-axis precision motion stage to sweep a collector plate over the emission region. The aforementioned authors either utilized hollow capillary needles or externally wetted needles and traditional electrospray for their studies. Because the ILFF electrospray technique studied here is intrinsically different than both needle and capillary spray of conventional liquids the beam divergence must be quantified experimentally. In particular, the strong axial magnetic field intrinsic to the technique coupled with the superparamagnetic nature of the propellant may cause interesting and unexpected divergence behavior.

The goal of this study was to characterize the influence magnetic and electric fields beam divergence. To achieve this goal, an apparatus was constructed that can position a collector plate at a specific angle such that the local emission current can be measured for a single ILFF emitter.

II. Experimental Setup

Testing was performed within the Ion Space Propulsion lab at Michigan Technological University. The following sections will describe the testing apparatus used to measure emission angle, the ILFF used during the testing, and finally, the testing procedures.

A. Testing Apparatus

A diagram of the single tip emitter is presented below in Figure 2. An aluminium reservoir (1) supports the ILFF (2), and magnets (3) cause the ILFF to form a single peak via the Rosensweig instability. When the fluid reservoir is biased with respect to the grounded extractor electrode (4), emission occurs. A 4-mm-diameter aperture (5) was centered over the reservoir and emission current was measured by a collector downstream of this aperture (not shown). The extractor plate and top of the fluid reservoir were separated 6 mm using PTFE spacers (6). For two and three magnets, the field at the top of the fluid reservoir was measured to be 115 and 230 gauss, respectively, with no

Figure 1: Representative ferrofluid peak with magnetic field shown with a US penny for scale.

fluid in the reservoir. The fluid reservoir was filled such that a 3 mm tall peak was formed.

To measure the angular current distribution, an emission mapping apparatus was constructed, shown in Figure 3. A stepper motor (7) connected to a positioning arm (8) sweeps a collector (9) over the emission region to measure current. The collector was the exposed face of a 2.35-mm-diameter tungsten rod that was insulated along the sides. The distance between the top of the fluid reservoir and the collection surface was 19 mm. A hard-stop (10) provide a datum for each mapping event. A limit switch (11) was utilized to determine the end of test location while an optical rotary encoder (12) measured angular position.
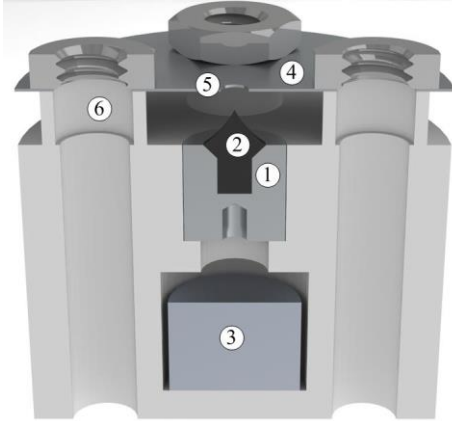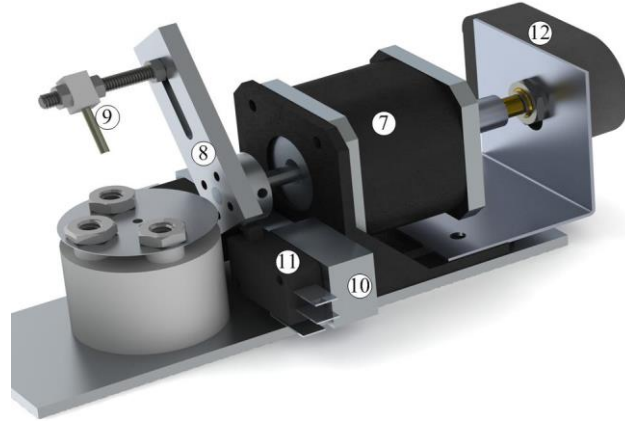

Figure 2: Single tip ILFF emitter.

Figure 3: Emission mapping apparatus.

A SureStep STP-MTR-17040D double shaft stepper motor was used to drive the current collector. The motor has a resolution of 200 full steps/revolution; when half stepping is commanded the resolution becomes 0.9°/half step. An initial angular alignment was obtained by driving the collector arm into a hard stop which was 90° off axis of the emission centerline. A US Digital S5 differential output optical shaft encoder was used in conjunction with a US Digital Quadrature to USB adapter to measure the shaft angle.

B. Ionic Liquid Ferrofluid

The carrier fluid for the ILFF utilized during this testing was EMIM-NTf2 (1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide). When magnetic nanoparticles were added, a final solution was obtained that was 22% by weight nanoparticles. This ILFF was developed by Brian Hawkett and Nirmesh Jain at the University of Sydney in collaboration with Michigan Technological University.

C. Testing Procedures

The vacuum chamber in which this testing was performed had a base pressure of 5×10^{-6} Torr and pressure didn't exceed 2×10^{-5} torr during testing. All measurements were taken at 1000 Hz unless otherwise noted. For the emission mapping experiments, 100 current measurements were taken at each stepper motor location. Current was measured with a FEMTO DLPCA-200 precision current amplifier. A Matsusada AMT-5B20 high voltage amplifier was utilized which was coupled with an 33120A waveform generator to control the extraction voltage of the emitter.

III.   Results and Discussion

Experimental characterization of the single tip ILFF emitter included emission current vs. extraction voltage (I-V) characteristics and beam divergence measurements for two different magnetic fields and three different extraction voltages.

A. I-V Characteristics

To obtain I-V traces for the emitter, the ILFF reservoir was biased using an alternating polarity high voltage amplifier. A signal generator was utilized to control the extraction voltage waveform. The polarity of the bias was controlled using a 100 mHz square wave which was amplitude modulated by a 200 mHz ramp function. An example bias voltage trace is presented in Figure 4:

Prior to each set of beam divergence measurements, an I-V trace was generated. From this trace, the onset of emission voltage was determined to establish the relevant voltages to be used during beam divergence mapping. An overlay of the 2-magnet and 3-magnet IV traces are presented below in Figure 5:
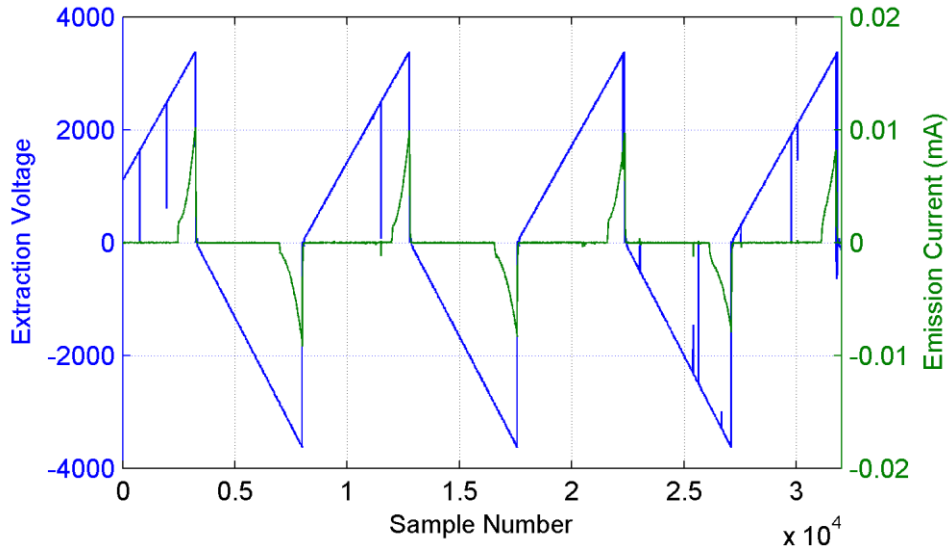
Figure 4: Current and Voltage plotted against sample number for EMIM-NTf2 I-V characterization with 2-magnets.
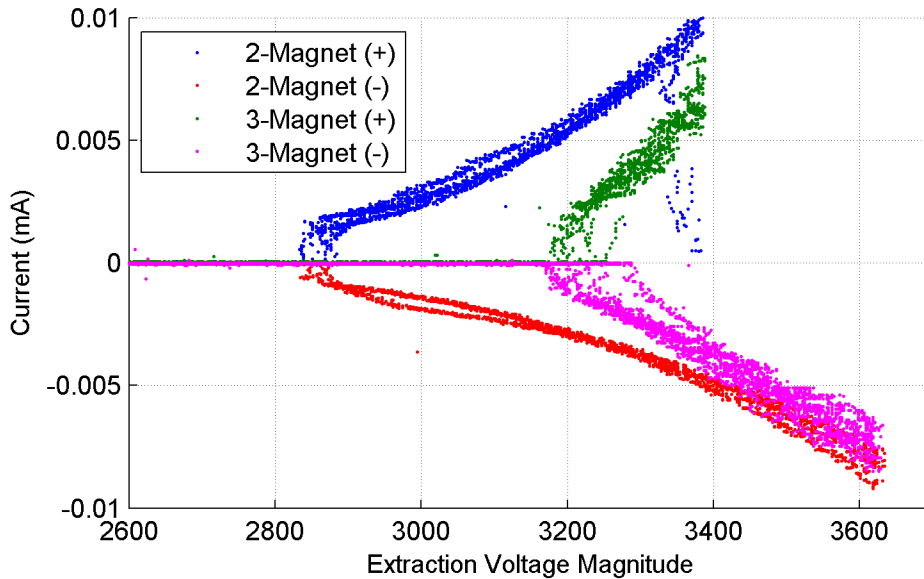

Figure 5: Current and Voltage relation for EMIM-NTf2 emission with 2-magnets. It should be noted that the extraction voltage magnitude is presented on the x-axis.

From the above plot it can be observed that the IV traces for both configurations do not overlay on another. This could be a result of several factors, which include: (1) different initial ILFF volumes within the reservoir, (2) different initial peak heights, and (3) the differing magnetic stress on the fluid. Due to the viscosity of the ILFF, precise volumetric samples could not be obtained with a micro pipette or syringe. Therefore, initial peak height was utilized as a common factor between trials. Unfortunately, a large degree of uncertainty was present in this method.

The onset voltage for the 2-magnet case was measured to be 2850 V. Somewhat arbitrarily the voltages chosen for emission mapping were 3000, 3200, and 3400 V. After these tests, the fluid reservoir was refilled and an IV trace was taken for the 3-magnet case. Since the same extraction voltages could not be reused, it was decided to record the lowest-voltage emission map at the condition that produced roughly the same current as the 2-magnet 3000 V case. As shown in Figure 5, this corresponded to 3200 V for the 3-magnet case. Emission maps were also taken at 200 and 400 volts above this datum.

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B. Emission Mapping

Emission mapping was first performed for the 2-magnet configuration. The desired voltage was applied to the source and the Faraday probe was swept from –90 to 90 degrees over approximately 25 seconds. The source voltage was then removed. Sweeps were made with 3000, 3200, and finally 3400 V. This sequence was then repeated such that a total of three traces were obtained for each of the three voltages. The same process was followed for the 3-magnet configuration. An overlay of the three traces for the first collection set of the 2-magnet configuration is presented below in Figure 6 with the 3-magnet configuration in Figure 7. The shaded region around each line represents an uncertainty of ± 1 standard deviation.
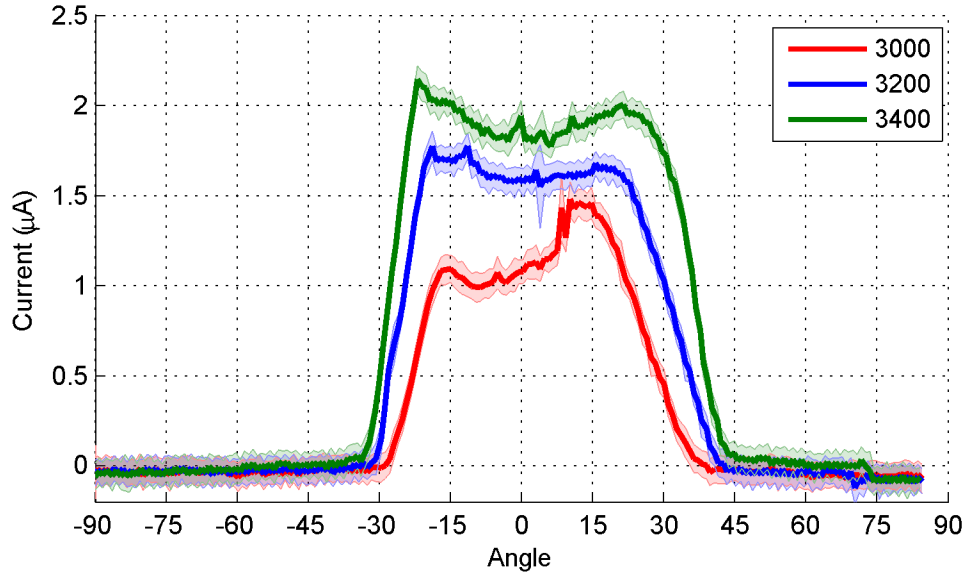

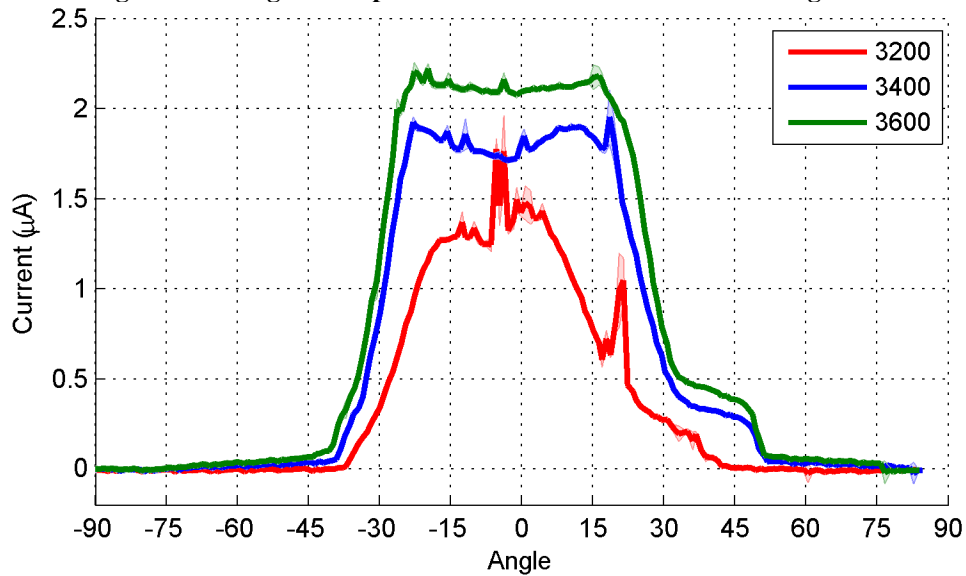**Figure 6: Divergence map for EMIM-NTf2 emission with 2-magnets.**

Figure 7: Divergence map for EMIM-NTf2 emission with 3-magnets.

Three emission maps were recorded at each extraction voltage. It should be noted that the emission profile varied from case-to-case despite using constant voltage, as can be seen in Figure 8. Because the setup did not permit measurement of total beam current while emission maps were being recorded it is not known whether the source operation was identical for each test or even constant during a single test. In other investigations with ILFF electrosprays it has been noted that the emitter tip visibly changes shape, a behavior that might account for the

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varied effects seen here. The tip variations may occur on a timescale faster than the 25 seconds required to complete a single emission map, thus temporal variations might be convolved into angle ordinate of each curve.
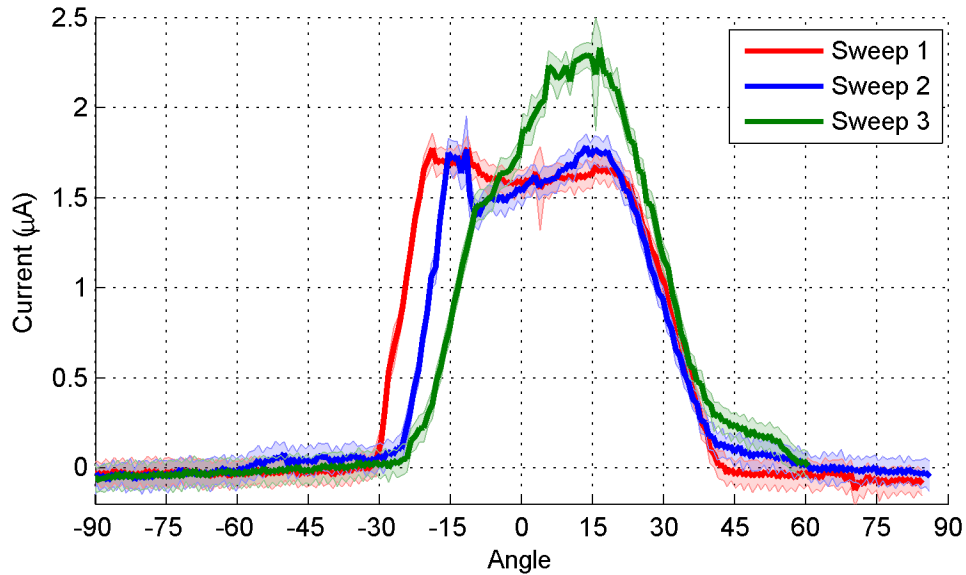

Figure 8: Divergence map for EMIM-NTf2 emission with 2-magnets and fixed extraction voltage of 3200 V.

Finally, presented in Figure 9 is a comparison of the 2- and 3-magnet configuration for approximately equal emission currents, obtained from the IV trace.
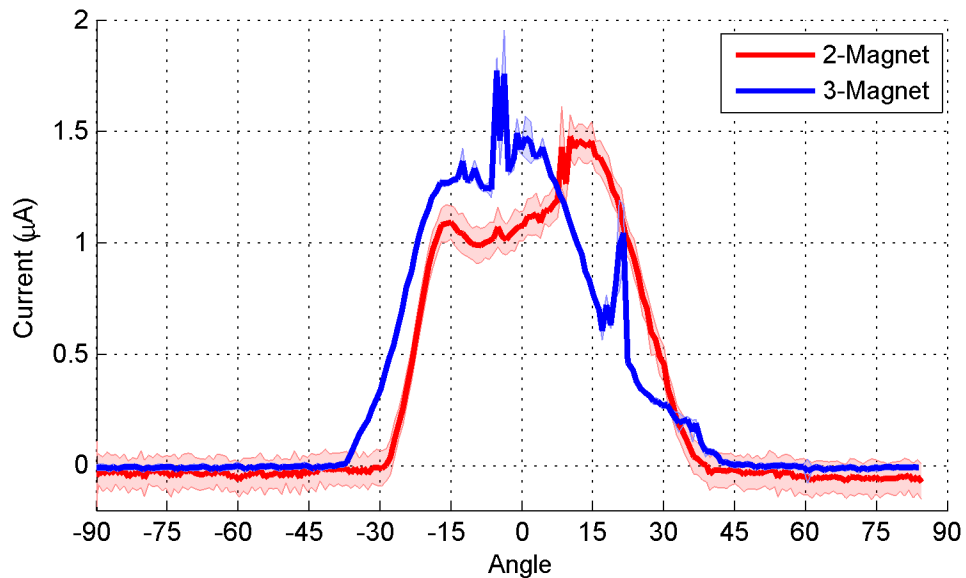

Figure 9: Divergence map of EMIM-NTf2 emission with 2- and 3-magnets for same IV emission current. The 2- and 3-magnet profiles were taken at 3000 and 3200 V, respectively.

C. Stability Testing

A longer duration test was performed to determine the stability of the emission at constant voltage. This was performed by applying an alternating extraction voltage to fluid in the form of a 250 mHz square wave with an amplitude of ±3.5kV. It is evident that, although the emission current shows a slow overall decline, the timescale of this change is long compared to the 25 seconds required to record a single emission map. However, random

transients are seen to occur on a faster timescale that could be responsible for spurious changes in the measured angular patterns.
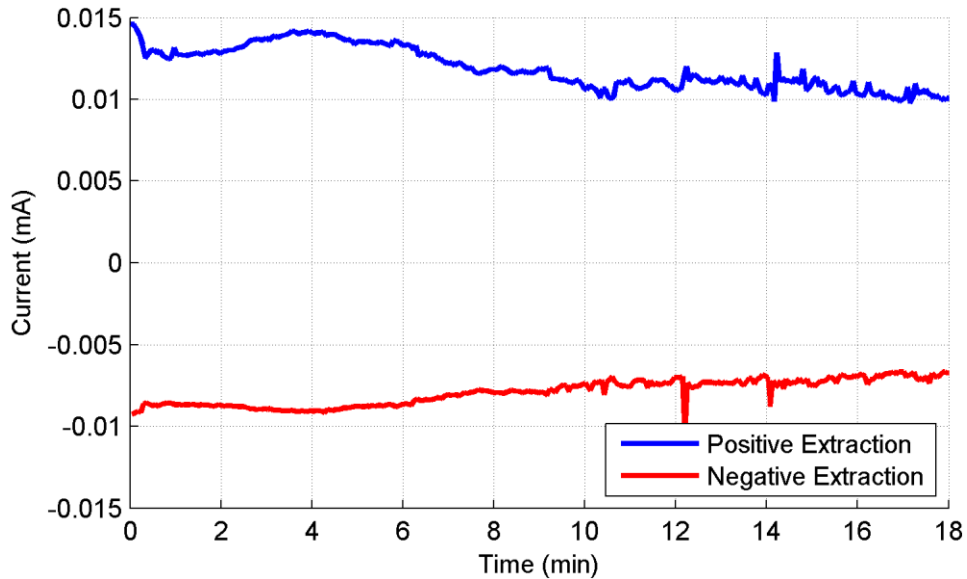

Figure 10: Long duration emission test with alternating extraction voltage. The ILFF peak was observed to release steady current for an additional 30 minutes (not shown) before any considerable drop in current was observed.

One particular case of interest was observed during the two magnet testing was a double peak emission profile, presented below in Figure 11. The physical phenomenon that formed in this profile cannot be confirmed. However, in previous studies where the emitter was placed in front of window to enable photography, instances have been observed where two emission sites formed on the ferrofluid peak. An example of one such instance is included below in the following figure.
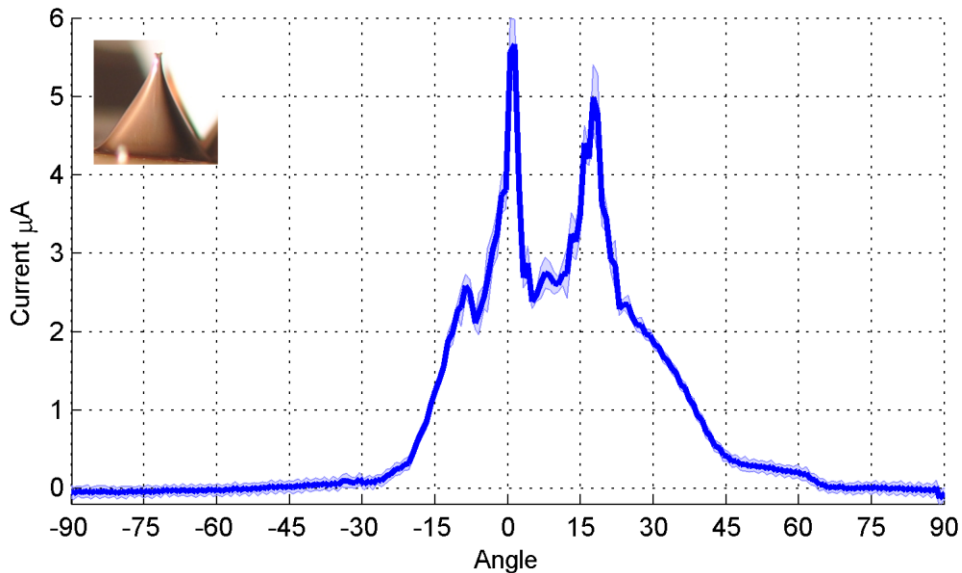

Figure 11: Double peak emission profile observed during emission with 2-magnets and fixed extraction voltage of 3400 V.

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IV. Conclusion

A single tip ILFF emitter and emission mapping apparatus was constructed using a ferrofluid composed of magnetic nanoparticles suspended in EMIM-NTf2. Using the apparatus, the emission current and extraction current-voltage characteristic was measured. The angular current distribution was then determined when both two and three magnets were used to form the ferrofluid peak. Finally, a long duration emission test was performed to assess the stability of the ferrofluid emitter.

It was observed that the width of the emission profile for the 2-magnet case was less than that of the 3-magnet case by approximately 10 degrees. With regards to the impact of extraction voltage, no significant correlation was observed in either the 2- or 3-magnet case.

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