

INDUCTIVE MICRO-FLASH DESORPTION ($I_{\mu}FD$) FOR A MOLECULAR-LEVEL UNDERSTANDING OF FUEL LUBRICITY

TRACK OR CATEGORY

Lubrication Fundamentals

AUTHORS AND INSTITUTIONS

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INTRODUCTION

Liquid fuels aboard modern high-performance aircraft currently fulfill the role of not only the propellant, but also a heat transfer fluid and a hydraulic fluid. The fuels themselves have now reached their thermal capacity for effective cooling, and any additional heat load results in unfavorable thermal stress to the fuel, restricting further performance gains. A proposed method to improve the operability of these aircraft and increase efficiency is to eliminate the entire lubricant system and require that the fuel serve not only as the propellant and coolant, but also as the lubricant [1,2]. To enable this transition, it is necessary to identify the important characteristics of fuel lubricity in order to design fuel blends that can optimize this function. In this work, we aim to demonstrate a method for quickly identifying potential lubricant candidates within complex mixtures.

Fuel pump failures in jet aircraft due to severe hydrotreatment processes to remove sulfur from the fuel spawned research to determine the classes of molecules that enhance or reduce the fuel's lubricity [3-6]. In more recent years the ever more stringent sulfur specifications for diesel fuels continue to make this an active area of research [7]. Such classes of molecules that have shown to improve lubricity include alkyl polar compounds (ex. fatty acids), phenols [5,6], certain nitrogen-containing species [4], and aromatics [3,8]. In general, it is understood that the molecules attributed with lubricity are surface active, forming a thin, protective film on the surface. Since they are only required in trace amounts ($< 0.1\%$) in order to be effective [9], it can also be difficult to detect and identify exactly what species are important.

At NIST we are developing a fast, solvent-free extraction technique called Inductive Micro-Flash Desorption ($I_{\mu}FD$) to characterize the molecular species in complex mixtures that are involved in boundary lubrication through direct measurement. The technique takes a sample immersed in a fluid that contains surface-active species and rapidly thermally desorbs the interacting species from the surface with induction heating, as depicted in Figure 1. The desorbed species are then detected by suitable analytic techniques such as gas chromatography with mass spectrometry. In this paper, attention is focused primarily on the experimental methods employed, with only a brief discussion of preliminary results.

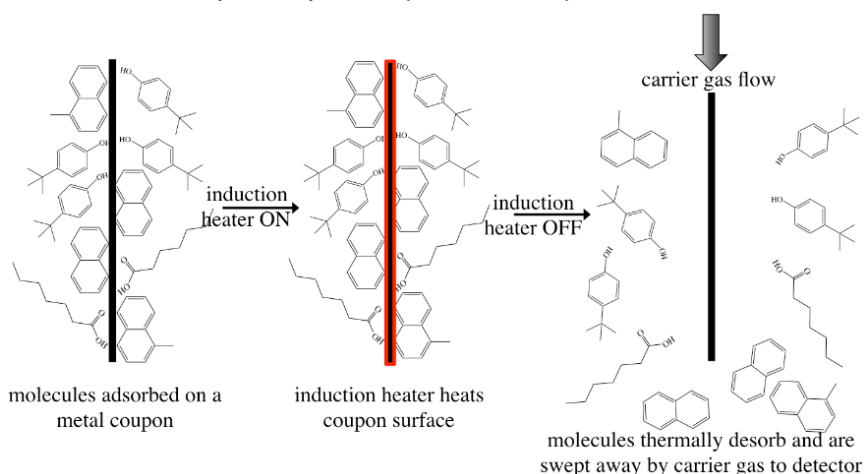


Figure 1: Inductive Micro-Flash Desorption ($I_{\mu}FD$) for trapping and sampling molecular species that provide lubricity.

EXPERIMENTAL APPROACH

There are several methods for characterizing lubricity, from tribometers that measure friction coefficients and wear rates at the macroscale, to nanotribology with an atomic force microscope to examine friction on the nanometer scale [10]. The basis of this research assumes that in order for something to provide boundary lubrication, it must be surface active [8], as shown in Figure 2. We therefore, must identify in a liquid sample just what species interact with various surfaces to provide lubricity. The concept is to immerse a wire coupon of suitable material in a mixture that may or may not contain surface-active species. That coupon is typically a metal, since metals comprise mechanical systems that wear and require lubrication. After a suitable immersion period at a selected temperature and pressure, the sample is removed from the solution and any species that have an affinity for the surface will remain on the coupon. What is required for evaluation is a nearly instantaneous release of the surface active species from the coupon, without the use of a solvent that can interfere or cause contamination. In order to achieve this rapid and clean release, we developed an induction heating device, coupled with a sample recovery and analysis metrology to remove surface active species.

The method and apparatus we developed is called Inductive Micro-Flash Desorption, $I_{\mu}FD$. Heating by induction requires a high-frequency resonant circuit. The high-frequency alternating current through the work coil produces a high-frequency alternating magnetic field within the vicinity of the coil, as shown schematically in Figure 3. When an electrically conducting material (the workpiece/coupon) is placed within the coil, the field induces a current in the coupon that primarily heats the material's surface [11,12].

By use of induction to heat the coupon, the adsorbed species are removed without a solvent. In the case of a mixture that contains unknown adsorbents, the choice of solvent to remove the adsorbed species would be ambiguous. If there are species that are unable to dissolve into the solvent, they will not be detected. In addition, the solvent could potentially interfere with the detection method (in this case, chromatography). For this reason, we have chosen a solvent-free extraction technique. The $I_{\mu}FD$ technique provides rapid, non-contact heating, keeping the sample free of possible contaminants and in an inert environment.

The high-frequency power supply required for induction heating and some of the important components are shown in Figure 4a. The MOSFETs amplify the 12 V, 5 A input signal from the DC power supply to produce a voltage on the coil in excess of 100 V. The resonant frequency of the circuit is approximately 100 kHz, which is sufficient to heat relatively 'poor' electrical conductors (high electrical resistivity) such as room temperature steel, but the critical frequency required for better electrical conductors (such as copper), is much higher for the same workpiece diameter [12]. For this reason, the resonant circuit may require modifications for testing certain surfaces. The coil, shown in Figure 4 is made of 1.8 mm copper tubing and is 2 cm in diameter, 3 cm in length, with about 10 turns of the tubing. The coil was coated with a thermally conductive ceramic to protect and provide stability to the coil; the ceramic does not interfere with the magnetic field.

The injection sampling procedure with the wire coupon and syringe assembly is shown in Figure 4b. The coupon used for proof-of-concept tests was a 400 series stainless steel (magnetic), 0.5 mm diameter and about 30 mm long. The coupon was immersed in diluted solutions (0.05 to 10 wt-% in *n*-hexane, *n*-decane or *n*-dodecane) of a jet fuel (JP-8), a diesel fuel surrogate (9 components), JP-8 with a high-temperature additive [13] or a fully qualified lubricant for aviation turbine engines (polyol esters with tricresyl phosphate for surface passivation). Immersion times of the coupon in the liquid ranged from 10 min to 2 hr. Upon removal from the liquid mixture, the coupon was gently blown dry with compressed air to remove any residual solvent and sealed inside the gas-tight syringe. A flow of either helium or carbon dioxide (for improved collisional

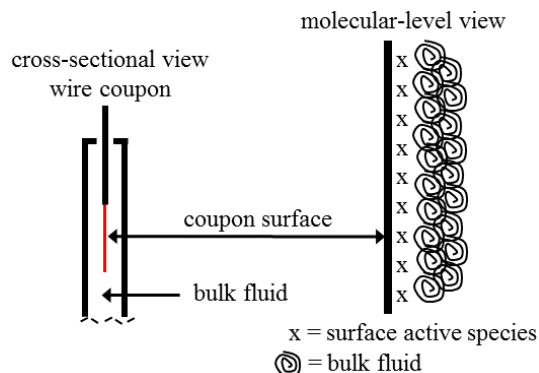


Figure 2: Surface-active species that provide lubricity will remain on surface; measured concentrations may differ from the bulk fluid.

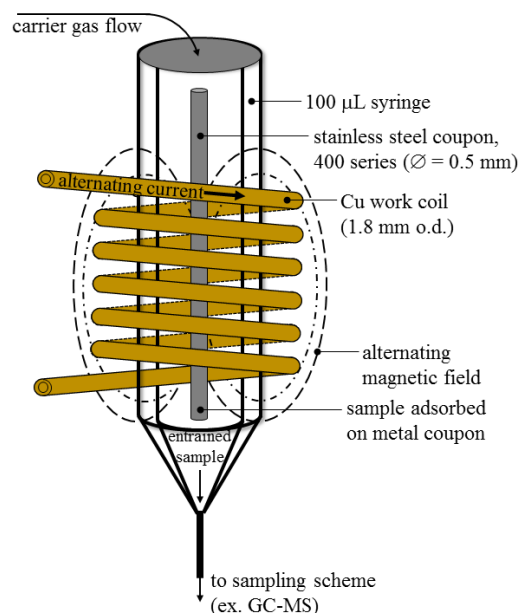


Figure 3: Heating the wire coupon by induction to remove adsorbed species.

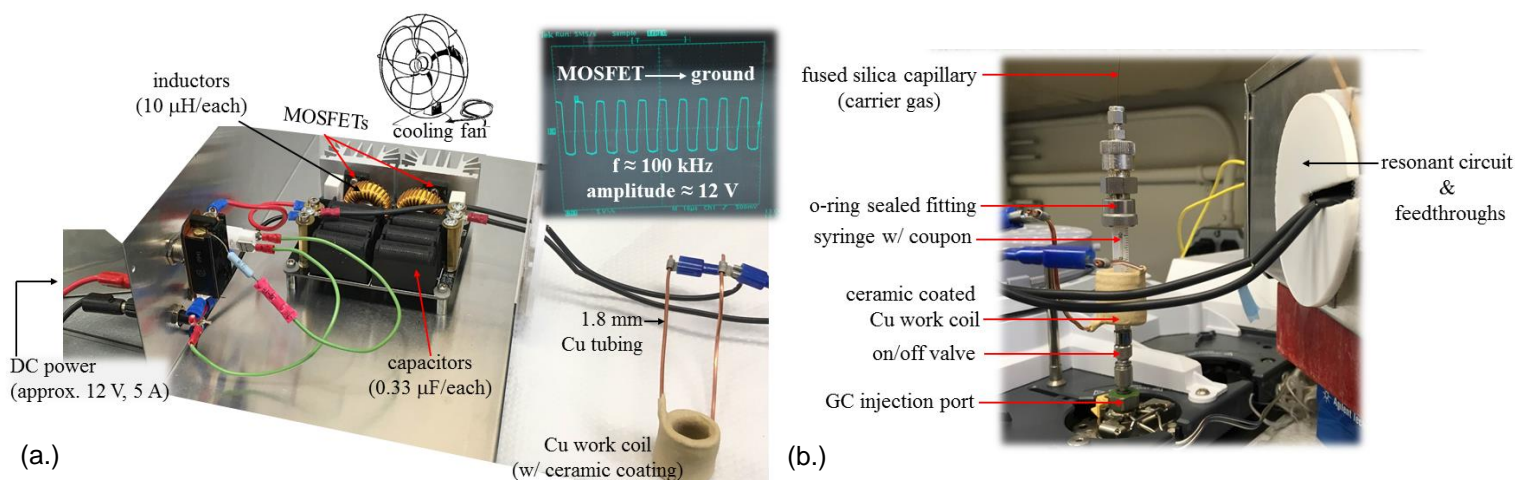


Figure 4: (a.) The resonant circuit and coil used for induction heating. (b.) Sampling method to GC-MS.

efficiency/affinity to sweep the species away from the heated coupon) was sent through the syringe containing the coupon. The flow began 5 s prior to heating and continues for 30 s after heating. In order to heat the coupon, the induction heater was pulsed on for 5 s. Based on inductively heating the 400 series stainless steel coupon in air, the resulting color of the oxide coating is a yellow-brown, indicating a relatively thin oxide layer was formed. At this time, we have not fully characterized the temperature profile of the heating event; however, measurement of the surface temperature is underway.

Preliminary results show that the amount of substance detected increases with time immersed in solution, indicating that time for surface-active species to diffuse to the metal coupon is an important parameter. Results also indicate that species that have an increased affinity for the surface are present in higher relative concentrations on the coupon than they are in the bulk fluid. An example of JP-8 in the bulk fluid versus JP-8 sampled from a stainless steel coupon is shown in Figure 5.

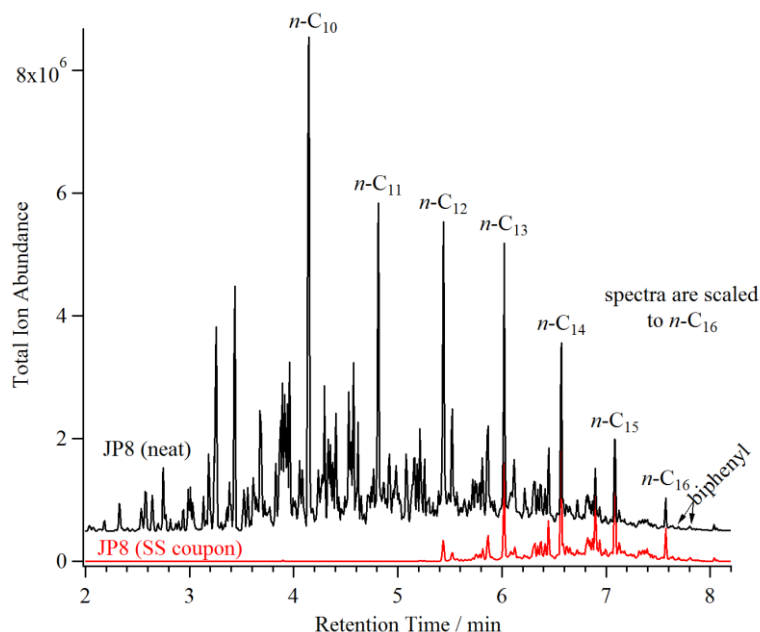


Figure 5: Total ion chromatogram (GC-MS) of a neat JP-8 sample compared to species desorbed from a stainless steel coupon immersed in solution

CONCLUSIONS

We have developed a fast, solvent-free extraction technique to characterize the molecular species in complex mixtures that are involved in boundary lubrication. Preliminary results demonstrate that the Inductive Micro-Flash Desorption technique is an efficient method for identifying surface-active species in fuels and lubricants. We also anticipate that other potential uses of this device could include biological applications (implants/sensors/microbial-induced corrosion) and forensic artifacts.

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KEYWORDS

Boundary Lubrication: Boundary Lubrication (general)
Lubricants: Jet Engine Oils
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