Synthesis and characterization of thermal properties of a micrometer sized Graphite-$FeCl_3$ intercalation compound

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During the last few years graphite intercalation compounds have attracted a great deal of attention, particularly because of their promise as materials of high conductivity and anisotropy, especially Graphite-$FeCl_3$ intercalation compound (GIC-$FeCl_3$) owing to its extensive usage in high density lithium ion Batteries and other commercial applications. Various research has been published that experimentally investigates its physical and electronic properties from battery perspective. But what's globally missing is a critical investigation of its nanoscale thermal properties mainly temperature and size dependance of a) in-plane and cross-plane thermal conductivity b) thermal boundary conductance c) heat capacity, etc. Herein, we demonstrate first ever step towards fabrication and characterization of thermal properties of micrometer sized GIC-$FeCl_3$. Synthesis is achieved by first mechanical exfoliation of HOPG onto a Si/SiO_2 substrate using intercalation setup that realizes one-zone vapor transport method at 340°C and 1atm Argon pressure to produce micron sized GIC with every 3rd graphite layer intercalated with $FeCl_3$ on its both sides. This characteristic layer ordering phenomena is determined using Raman spectroscopy (Fig 1). Next, for thermal property characterization of such thin and micron sized graphite intercalated flake, a powerful and versatile ultrafast laser-based time-domain thermoreflectance (TDTR) as shown in will be employed to accurately determine in-plane and cross-plane thermal conductivity, thermal boundary conductance (between flake and aluminum) and specific heat from just one set of measurement, which is what makes TDTR, a highly powerful and robust thermal characterization technique. However, to deposit aluminum on a micrometer scale flake is yet another challenging task which is accomplished using laser lithography microfabrication followed by ultra-high vacuum aluminum deposition as illustrated in fig 3. Once aluminum deposition is done, TDTR will be employed in future and measurements will be performed at different temperatures for different stages and sizes of intercalation compound, with the goal of exploring thermal transport in GIC-$FeCl_3$. Atomic force microscopy has been employed to determine thickness of intercalated flake from which interlayer spacing expansion can be calculated.

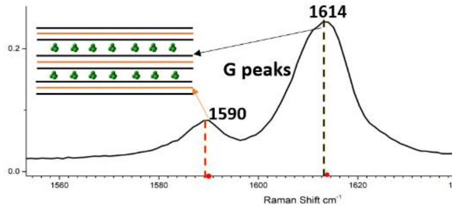

Fig 1: Measured Raman spectroscopy peaks: G1peaks (1590cm^{-1}) originating from interior graphite layers(shown in orange) ,and, G2 peaks (1614cm^{-1}) from intercalated layers(shown in black in cartoon)

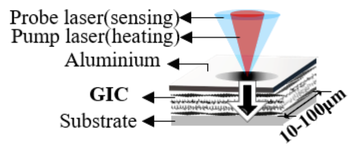

Fig 2: TDTR laser configuration for probing thermal conductivity. Downward arrow shows heat flow in downward direction

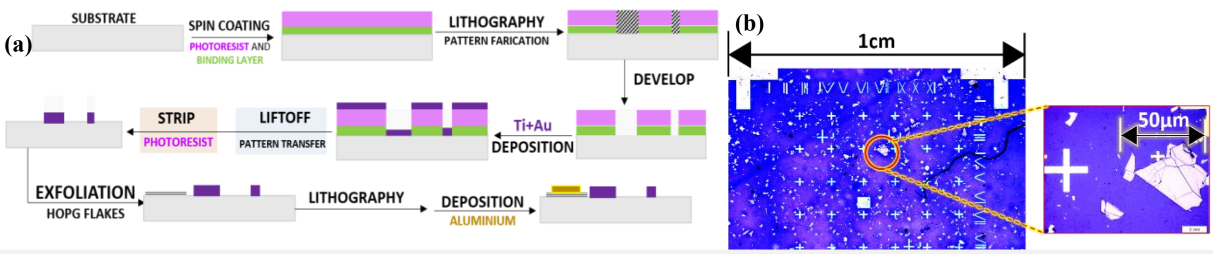

Fig 3: (a) Microfabrication using laser lithography technique to generate high resolution pattern to precisely locate a micro-sized intercalated flake's location as shown in (b) and then performing lithography of same pattern again on same substrate for deposition of aluminum for subsequent TDTR characterization of thermal properties of interest