Impact Case Study

UoA 13: Electrical and Electronic Engineering, Metallurgy and Materials

Bringing nanomaterials research to industrial production

Nanomaterials research at Ulster into materials including diamond-like carbon (DLC) ultra-thin films, carbon nanotubes (CNT), graphene, silicon and metal oxide nanoparticles has resulted in direct uptake by major industrial manufacturers and led to a directly quantifiable socio-economic impact via added value, improved efficiencies and cost-savings and has secured or increased the employment of skilled engineering staff. Examples of this impact since 2008 include ceramic nanoparticles research in partnership with AVX Ltd that resulted in improved production efficiency processes (up 20%) and higher quality devices (up 10%). Research into ultra-thin DLC films, funded by Seagate, has led to their incorporation into magnetic media. Our nanoparticle research has attracted a new spin-in company SiSaf Ltd. (2009) and by incorporating NIBEC's expertise in nanomaterials into its business plan, the company was able to grow to a valuation of £3.5m and employ 7 people in skilled technical positions.

The Nanotechnology R&D activities within NIBEC have a coordinated focus on developing a fundamental understanding of how materials processing impacts on the resultant characteristics and properties of ultra-thin films (<2nm), nanoparticles and surface properties at the nanoscale. We have an established track record in the preparation, analysis and processing of materials including diamond-like carbon (DLC), carbon nanotubes (CNT), graphene, and various nanoparticle systems (Si, group IV alloys, ferroelectric ceramics, and metal oxides). This area has received substantial support from EPSRC, the Royal Society, the EU Framework Programmes (FP4 – FP7) and InvestNI in collaboration with and/or through direct funded from industry (Seagate, AVX, SiSaf, Intel, Glaxo-Smith-Kline, Schrader Electronics, Analog Devices, TFX Medical, Medtronic, Labcoat, Kelvatech, Randox). The strategic aim of this research is to advance the understanding of nanomaterials for device fabrication and to promote its integration into advanced industrial manufacture. The associated publications highlight our expertise in fabrication & advanced characterisation that led to direct industry impact.

A number of targeted equipment grants have led to the creation of one of the most advanced academic nanomaterials research facilities in Europe (£15m of capital investment in the last 10 years from SRIF, RCIF, DEL, EPSRC)[6]. Recent highlights include novel equipment for the creation of ultra-thin carbon/CNT/graphene films, a nanoparticle production laboratory and advanced characterisation based on our new HRTEM. Our fundamental strengths have centred on; (i) advancing metrology for measuring nano-scale hardness, film thickness, internal stress and adhesive strength on ultra-thin (1–50nm) carbon layers; (ii) nanoparticle fabrication (via laser, microplasma, milling and wet chemical synthesis) and characterisation of both the materials and the synthesis processes; (iii) nano-scale surface-engineering and functionalisation with associated high-resolution analysis (HRTEM, ToF-SIMS, XPS). The recently established £2m nanoparticle laboratory is key to the synthesis, characterisation and understanding the unique properties of dispersed nanoparticles and it has attracted industry (AVX, Bombardier, SiSaf and Randox) to support our research.

For material synthesis, our primary strength is in plasma-based deposition, surface engineering and functionalization. We have at hand a large number of plasma systems operating from DC up to microwave frequencies and from vacuum to atmospheric pressure. We host advanced pilot-scale industrial systems supplied through collaboration with equipment manufacturers. These include a custom-built microwave plasma system (Seiki, Japan), which was the world's first system for the deposition of highly oriented nanotubes and a FCVA - filtered cathodic vacuum arc system (NTI Technologies, Singapore) for the deposition of hydrogen-free highly diamond-like carbons for Seagate. The research groups involved have also designed and constructed a suite of advanced multi-functional plasma systems in-house, including pilot (reel-reel) production systems with integrated specialist diagnostic tools for concurrent measurement of plasma and material properties during the growth of nanoparticles. This facility is being further developed towards providing low-pressure and atmospheric pressure capabilities for general large area/low cost processing and precision three-dimensional coatings for medical implants. We have four patents awarded and active commercialisation in progress. Recently a University of Ulster spin-out company Surf Spec Ltd was established (BM) to exploit aspects of plasma surface modification as they apply to biomaterials. In-house materials characterisation is complemented by access to international synchrotron (Daresbury, Diamond, CERN) and neutron diffraction (Rutherford Appleton) facilities via numerous CCLRC and EU awards.

We have secured funding for 15 projects related to nanomaterials and of these, two major research awards, totalling £1.6m since 2008, involve collaboration with AVX Ltd (TB, PL). The research aims to link fundamental ceramic nanoparticle properties to industrial processing conditions. The resultant impact on the electrical and reliability characteristics of multi-layer capacitor devices has allowed the company to develop a new generation of high value products. Core elements of this work along with the processing expertise developed are now also being targeted at the development of ceramic/clay fillers for Bombardier.

SiSaf Ltd are a 2009 spin-in company with NIBEC staff on the board as CSO/Director. A collaborative research venture with NIBEC aims to produce and functionalise nanoparticle silicon for use in drug and therapeutic transdermal delivery applications. This work relies on the provision of high-quality and precisely controlled 50nm particles of Si with defined porosity/surface properties in order to allow specific functionalisation and drug attachment. This has involved in-vitro testing of Si particle nano-toxicity at the NIBEC Cell Culture Facility. The data has provided the platform for a vitamin C dissolution study in an animal model which indicated that the SiSaf delivery system is 30% better than a standard drug delivery system. Current studies are examining the use of nano-silicon for transdermal therapeutic delivery involving a USA based human clinical trial. SiSaf won the 2010 Award for Most Promising Technology at the Irish Technology Leadership Group (ITLG) awards ceremony in Silicon Valley, USA.

The majority of the carbon-based ultra-thin film and nanoparticles work commenced in 2000 arising from the burgeoning global interest in diamond-like carbon (DLC), carbon nanotubes (CNT) and graphene for electrical, optical and mechanical applications. Our research was guided in the main via company initiated collaborations with Seagate Technology, Daewoo Electronics and TFX Medical. Each company had a particular requirement for ultra- thin, low friction, dense (pin-hole free) and durable films of 1 nm (Seagate) – 20 nm (TFX) in thickness. The experience gained in delivering the challenging specifications set down by these globally leading companies provided the model for the NIBEC technology transfer process and it is currently focussed on the controlled growth and properties of CNT arrays for biomedical and biosensor applications. Original work in the application of CNTs has led to the development of inventive electrochemical platforms for bio-sensing applications, several of which are now patent protected. Also a spin-out company, Graphene International Ltd (2012) has recently been established by PP with patents and licenses in the area of graphene production scale-up.

Ulster has had on-going links with AVX Ltd for over 25 years with the company funding research, providing PhD studentships and offering industrial support for numerous collaborative R&D projects. Most recently (2009), NIBEC and AVX commenced a large scale project to improve the quality of the dielectric layers in the Multi-Layer Ceramic Capacitors (MLCC) that the company produce in millions of units per day. This project was highly successful with major improvements in nanoparticle dispersion which the company have now fully integrated by into their volume production lines (including Czech Republic). This has had a major impact upon product development and business results. Building on the success of this collaboration, in 2012 AVX co-funded a second project in collaboration with NIBEC specifically focusing on improvement of the base metal electrode (BME) system utilising nanoparticle nickel for MLCCs. This project has already identified improvements in BME ink formulation and processing. This has arisen due to the direction and outputs achieved from the materials characterisation and the understanding associated with electrical characterisation of both raw materials and devices. Improved control of de-agglomeration and dispersion of barium titanate powders and metal oxide dopants along with feedback from nanoscale measurement and characterisation data (impedance analysis, dispersion and binder-particle interaction) have allowed the company to greatly enhance their manufacturing processes. A third and much larger project (£4m) has recently been submitted to Invest NI (July 2013) focusing on defect control within the dielectrics layers. Furthermore, the knowledge gained through our recent collaborative activities has allowed AVX (NI) to gain substantial credibility with USA customers such as NASA, BMW, Ford and the European Space Agency. New device characterisation and fabrication capabilities introduced by NIBEC include, Electrical Impedance Spectroscopy, FIB preparation of lamellae and HRTEM analysis, XPS and ToF-SIMs, particle size analysis (BET, electroacoustic and dynamic light scattering), new wetting and dispersion technology and a rare earth dopant programme. All of these interactions have impacted on international sales, growth, global market penetration and job sustainability. Joint publications, publicity and sponsored PhD programmes have resulted, leading to a strong and fruitful relationship.

SiSaf Ltd.: SiSaf is a spin-in to NIBEC and is developing drug delivery formulations that use a patented bioactive and non-toxic form of silicon nanoparticles (≤ 50nm) that are designed to allow compounds to penetrate into the deeper layers of the skin without adverse effects. Using the extensive knowledgebase acquired over the last 15 years in the area of nanomaterials, NIBEC is assisting the company to develop improved fabrication and characterisation techniques. NIBEC have a senior staff representative on the board as CSO (JMcL). SiSafs core fabrication and characterisation laboratories are based at NIBEC and with the investment of two joint £250k INI R&D grants, various consultancies and collaborative work, NIBEC and SiSaf have jointly developed a wide range of capabilities in silicon nanoparticle production. This work has led to an expansion of SiSaf to seven staff, increased laboratory space and enhancement of their patent, IP and knowledge assets. High quality silicon nanoparticles (50-100nm) are routinely batch-produced, functionalised, doped with drugs and characterised for trials and marketing samples to customers.

This interaction has already led to a better understanding of the fundamental properties of Si nanoparticles, its dependence of the formulation route and a more efficient processing protocol. The research has directly impacted on the valuation of the company with NIBEC staff producing key reports and know-how in a number of important areas, namely: the nature, size and surface characteristics of the 50 nm silicon nanoparticles; three technical reports on the scale-up viability for nanoparticle production via (i) arc deposition, (ii) via -milling and (iii) via Pulsed Laser Deposition (PLD) for production of smaller particles. Biocompatibility (and in particular nano-toxicity) has been investigated by NIBEC and a technical report issued which has impacted beneficially on investor confidence through our findings of non-toxicity which would have been a major barrier to full commercialisation. The company now has a valuation of £3.5m and is completing a set of £1.5m human trials in the USA to determine the suitability of the product for topical application. It has a Silicon Valley office at ITLG with John Hartnett as lead investor along with IUL, Crescent Capital and a set of private angels. The impact of having NIBEC team on board is significant and the following points were key to improving the company's overall value: nanoparticle fabrication (via plasma, CVD, wet etching and milling); nanoparticle characterisation via HRTEM, dynamic light scattering and zeta potential dispersion analysis, pharmaokinetics and nanotoxicity testing with human cell lines. In summary the company is in a strong position, with over £2m of investment to date and with Human Clinical trials near completion, Suzanne Siebert (CEO) has said "NIBEC's impact on the company has been critical, and as we develop even stronger international relationships in India, USA and throughout Europe, these engineers, who underpin our work are highly important partners".

SEAGATE: In 1994 the then core nanotechnology team at NIBEC, in conjunction with colleagues in QUB and local government agencies, were instrumental in attracting Seagate Technologies to locate a major production facility in the UK (in competition with South America, eastern Europe and Asia). The subsequent NIBEC relationships with Seagate R&D in both the UK and the USA were developed around nanoscale coatings of diamond-like carbon on a new generation of GMR read-write head devices. The NIBEC team utilised a novel plasma immersion technique called Filtered Cathodic Vacuum Arc (FCVA) that was developed in conjunction with Veeco Instruments (USA). This, along with our development of specific metrology protocols for analysing AlTiC ceramics, led to ultra-hard layers (30 GPa) with a credible measured thickness of 1.5nm, the world’s thinnest continuous carbon coatings. The ultimate result was a read-write head with a significantly increased iron content and a reduction in head-disc flying-height (due to the protective nature of the dense carbon layer). Seagate, in conjunction with InvestNI, invested over £5m in three key projects associated with the augmentation of this initial work, from 1996 to 2009. NIBEC and Seagate are currently planning a new programme of engagement in the area of Heat Assisted Magnetic Recording with a focus on our proven nanomaterials capability.