Institution: Sheffield Hallam University

Unit of Assessment: 13 Electrical and Electronic Engineering, Metallurgy and Materials

Title of case study: Advances in Physical Vapour Deposition based on High Power Impulse Magnetron Sputtering (HIPIMS)

1. Summary of the impact

Ehiasarian and Hovsepian of the Materials and Engineering Research Institute (MERI) have achieved significant **economic impact** through industrial uptake of their innovations in High Power Impulse Magnetron Sputtering (HIPIMS). Exploiting these innovations, HIPIMS treatments have been used by manufacturers to enhance the surface properties of millions of pounds worth of products. Applications include industrial blades, components within jet turbines, replacement hip joints, metallised semiconductor wafers and satellite cryo-coolers. Patents based on Ehiasarian and Hovsepian's research have achieved commercial success. In the REF impact period, HIPIMS machines equipped to deliver MERI''s HIPIMS surface pre-treatment have achieved sales of over £5m, and **income generated through SHU's HIPIMS-related licences has totalled £403,270**. In 2010 Ehiasarian's group established the **Joint Sheffield Hallam University-Fraunhofer IST HIPIMS Research Centre,** the first such Centre in the UK. This has broadened the industrial uptake of MERI's HIPIMS technologies and stimulated a network of sub-system providers.

2. Underpinning research

Hovsepian joined MERI in 1997 as a Senior Research Fellow in what was the then Surface Coatings Research Centre led by Prof Dieter Munz (submitted for REF2001, retired 2003). Hovsepian was promoted to a personal Chair in 2004 and assumed the Head of Research Centre role when Munz retired. Ehiasarian completed his PhD in the Surface Coatings Research Centre in 2002, was appointed to a permanent independent researcher role in MERI in April 2004 (he was entered in RAE2008 as an Early Stage Researcher on that basis) and promoted to a personal Chair in 2011. Both Ehiasarian and Hovsepian were submitted by MERI in RAE2008 and both have been at SHU throughout the REF period.

HIPIMS is a plasma vapour deposition (PVD) method for surface treatment and coating. It uses a powerful plasma discharge to generate an ionised vapour that is free of macro-particles. This vapour can be used either to pre-treat surfaces of target components or to deposit a range of coatings of specified composition and nanostructure. As detailed in **section 4**, surface pre-treatments and nano-scale coatings based on Ehiasarian and Hovsepian's HIPIMS advances have achieved a number of **economic impacts**, most obviously by enhancing the properties and utility of a range of end-products.

MERI's contributions to HIPIMS originate from Ehiasarian's thesis work (supervised by Munz and other permanent staff, and partially supported by [i]) which focused on the plasma physics diagnostics of what was, at the time, a very new branch of PVD. A specific innovation that resulted from this was a methodology for achieving "fully-dense hard" HIPIMS coatings [1,A]. This methodology, based on use of short bursts of very intense driving currents, proved capable of eliminating the intergranular pores commonly found in PVD coatings. Subsequent research performed within MERI between 2002 and 2012 addressed the fundamental issues of:

- HIPIMS coating microstructure, composition and adhesion [2.3,5];
- plasma pre-treatment of substrates [3];
- electrical gas discharge physics, plasma chemistry and distribution [4,6];
- and field-controlled plasma manipulation within the HIPIMS chamber [4].

This work demonstrated that previously unobtainable wear-resistance and coverage properties could be realised using appropriate combinations of electric and magnetic fields to manipulate the parent plasmas of different source materials [4-6,ii-iv]. Further research studied process-microstructure-performance relationships in HIPIMS-deposited thin films [2.3,5], particularly microstructure evolution and self-organisation phenomena.

In 2007 the group's HIPIMS machinery was up-graded to industrial production grade with an automated system. Uniquely, within the University sector, this provided an ability to develop the industrial applicability of HIPIMS innovations. This up-scaling proved to be critical in realising technology transfer to the manufacturing sector and, so, achieving international impact. In particular, it enabled the development of coatings for auto- and aero-engine components [v,vi],

cryogenic materials [vii], photovoltaic cell layers [viii], and biocompatible, antibacterial coatings [ix].

In RAE2008, the strong research quality of the MERI Surface Coatings Research Centre, under which this work was submitted, was specifically highlighted in the final HEFCE feedback. The academic and industrial reputation of HIPIMS research at MERI was further consolidated in 2010 with the establishment of the **Joint Sheffield Hallam University-Fraunhofer HIPIMS Research Centre,** partnered with the Fraunhofer Institute for Surface Engineering (Braunschweig, Germany) [B]. This was the first such centre in the UK. Ehiasarian is its Director. Also, in 2010, EPSRC supported the establishment of a joint centre between University of Leeds, University of Sheffield and MERI to establish a *"National Innovation Playground to Widen the Operational Envelope for Tribological PVD Coatings"* [iv]. Within this collaboration, Ehiasarian and Hovsepian contributed HIPIMS solutions to tribological problems for UK industry.

3. References to the research

[1] Ehiasarian, A.P., New, R., Munz, W.D., Hultman, L., Helmersson, U. and Kouznetsov, V., "Influence of high power densities on the composition of pulsed magnetron plasmas", *Vacuum* **65**, 147 (2002).
DOI: 10.1016/S0042-207X(01)00475-4 136 citations (WoS, Sept 2013)
KEY REFERENCE

[2] Ehiasarian, A.P., Munz, W.D., Hultman, L., Helmersson, U. and Petrov, I., "High power pulsed magnetron sputtered CrNx films", *Surface and coatings technology* **163-164**, 267 (2003)
DOI: 10.1016/S0257-8972(02)00479-6 129 citations (WoS, Sept 2013)

[3] Ehiasarian, A.P.; Hovsepian, P.E., Hultman, L., and Helmersson, U. "Comparison of microstructure and mechanical properties of chromium nitride-based coatings deposited by high power impulse magnetron sputtering and by the combined steered cathodic arc/unbalanced magnetron technique", *Solid Films* **457**, 270 (2004)
DOI: 10.1016/j.tsf.2003.11.113 88 citations (WoS, Sept 2013)
KEY REFERENCE

[4] Hecimovic, A., Burcalova, K. and Ehiasarian, A.P., "Origins of ion energy distribution function (IEDF) in high power impulse magnetron sputtering (HIPIMS) plasma discharge". *Journal of Physics D: Applied Physics*, **41**, 095203 (2008)
DOI: 10.1088/0022-3727/41/9/095203 25 citations (WoS, Sept 2013)

[5] Ehiasarian, A.P., Vetushka, A., Gonzalvo, Y.A., Safran, G., Szekely, L. and Barna, P.B., "Influence of high power impulse magnetron sputtering plasma ionization on the microstructure of TiN thin films", *Journal of Applied Physics*, **109**, 104314 (2011)
DOI: 10.1063/1.3579443 5 citations (WoS, Sept 2013)
KEY REFERENCE

[6] Ehiasarian, A.P., Hecimovic, A., De Los Arcos, T., New, R., Schulz-von der Gathen, V, Boke, M. and Winter, J., "High power impulse magnetron sputtering discharges: Instabilities and plasma self-organization", *Applied Physics Letters*, **100**, 114101 (2012)
DOI: 10.1063/1.3692172 11 citations (WoS, Sept 2013)

[i] EPSRC GR/R32420/01, PI Munz "Droplet Free Metal Ion Source for PVD Hard Coating Production" £76k (2001-03)

[ii] EPSRC EP/0049202/1, PI Ehiasarian "Fundamentals of High Power Impulse Magnetron Sputtering (HIPIMS) - Plasma Studies and Materials Synthesis" £190k (2006-09)

[iii] EU FP6, PI Hovsepian "CORRAL- Corrosion Protection with Perfect Atomic Layers" £61k (2008-11)

[iv] EPSRC EP/H050116/1 PI Hovsepian "National Innovation Playground to Widen the Operational Envelope for Tribological PVD Coatings" total £3.5m, to SHU £389k (2010)

[v] EU FP6 project, Hovsepian and Ehiasarian Investigators "*INNOVATIAL*" total €12.5M, to SHU €1.1M (2005-9).

[vi] *Mahle GmbH* (Brazil), PI Ehiasarian "Licence agreement for HIPIMS coating recipes for automotive engine piston rings" £50k (2013)

[vii] STFC *RAL*, PI Ehiasarian "Regenerator Technology for Stirling and Pulse Tube Cryogenic Coolers" £40k (2008-11)

[viii] EPSRC EP/J011398/1, PI Ehiasarian "High Efficiency $CuInSe_2$ Photovoltaic Modules Deposited at Low Temperature by HIPIMS" £329k (2012-15)

[ix] *Biomet* (UK/USA), PI Ehiasarian "Licence agreement for HIPIMS coating recipes, for use on

metal-on-metal hip implants" £300k (2012-14) £100k of which is in the REF period

4. Details of the impact

MERI's HIPIMS research has achieved **three streams of economic impact**: roll out of HIPIMS surface pre-treatment methods through coating system manufacturers; direct collaboration with product manufacturers; and discipline leading activities associated with the joint SHU-Fraunhofer HIPIMS Research Centre.

Economic impact by roll out of HIPIMS surface pre-treatment methods and other patents

In parallel with their academic research and publications, Ehiasarian and Hovsepian have developed a portfolio of patents to protect the IP in their HIPIMS innovations. Commercially, the most important of these has proved to be EP1260603B1 [A] (filed 2001, granted 2006, validated in 12 EU states), relating to the HIPIMS surface pre-treatment stage in the coating production process [1]. This patent has been monetised by equipping coating systems with special (licensed) modules that deliver the pre-treatment described in [A]. MERI's HIPIMS pre-treatment technology has been licensed non-exclusively in this way to four leading PVD coating system manufacturers: *Ionbond AG* (Switzerland) [C]; *SVS Vacuum Coating Technologies GmBH & Co* (Germany); *Hauzer Techno Coating AG* (The Netherlands) [D]; and *CemeCon AG* (Germany). This has achieved two classes of beneficiary: the coating system manufacturers, whose systems have been enhanced through incorporation of the MERI module; and system purchasers, who have acquired the capability of producing enhanced components. HIPIMS technology has become a key activity of *Hauzer Techno Coating* (~200 employees) [D] and *CemeCon* in the REF impact period.

Licence revenue paid to SHU (at a rate of €50k for entry to the programme and €25k for each sale or retrofit of a coatings system) explicitly links the system manufacturers and purchasers to the research described in **section 2**. In the REF impact period, six licensed systems have been manufactured and two more retro-fitted. These have generated more than £5m of sales to the manufacturer **beneficiaries** and **£238,089** of **licence income** to SHU. All licences (including those established before the REF period) will run through to expiry of the patent [A] in 2021. As a result, the operators of ten coating systems have been **beneficiaries** in the "system purchasers" sense over the REF impact period. Ehiasarian and Hovsepian have no direct interaction with the "system purchasers", so their specific uses of HIPIMS, and the associated sales figures, are in commercial confidence. However, typical applications are known to include wear and oxidation-resistant coatings for automotive engine components, resulting in longer lifespans and higher operating temperatures, and special coatings for dry high-speed machining tools, which yield enormous improvements in cutting-tool lifetimes and machining speeds. Each of the ten HIPIMS systems equipped with the MERI surface pre-treatment stage has the capacity to produce £10+m of components per year. Systems have been operating on two shifts per day to produce a range of high-value components [C,D].

In 2006 a further patent application was made to protect bias power supply designs developed within an industrial collaboration. This was granted in the UK (GB0607269B) and China (CN101461032B) [E] and remains in application stage in Europe, Korea and Japan. It is co-owned by SHU, *Hauzer Techno Coating* [D] and *Huettinger Electronic Sp z.o.o.* [F], with Ehiasarian and Hovsepian the leading inventors. This technology was commercialised in 2010, the two industrial co-owners retaining manufacturing and sales rights. Within the REF impact period, it has resulted in power supply unit sales exceeding £1m (**beneficiaries** *Hauzer*, *Huettinger* and their clients) and **licence income** to SHU of **£15,181k**.

Later patent filings by Ehiasarian and Hovsepian cover aspects such as differing coating types and technical advances such as high rate deposition, rare metal ion etching and inductively coupled etching. These have not earned any income in the REF impact period, but have been maintained within the overall IP portfolio due to their continued commercial potential.

Economic impact by direct collaboration with product manufacturers

Within the second impact stream identified above, Ehiasarian has delivered or started delivering collaborative and/or licensed research to a series of large manufacturers. In each case, **the manufacturer is a beneficiary** through enhancements made to their products. These include large **licence contracts** with *Mahle* **(£50k**) [vi] and *Biomet* **(£100k** pa for three years) [ix] to develop coatings for automotive piston rings and metal-on-metal hip implants, respectively. Additional collaborations yielding **economic impact through product enhancement** include:

- a £100k project funded by *OC Oerlikon Balzers* (2006-10), to develop HIPIMS coating

deposition technology to allow writing of miniaturised 3D-Integrated microelectronics devices. The company has integrated this capability into its Clusterline 200 system, a multi-stage coating machine, leading to sales exceeding €20m;

- a 2008-11 collaboration with the Space Science Technology Department of the *Rutherford Appleton Laboratory* (RAL) [vii], through which coatings developed by the Ehiasarian group overcame a long-standing issue with RAL's cryo-coolers for satellite applications. This work led to the satellites being judged flight worthy and superior to competitor technologies [G];
- a 2011 collaboration with *The Gillette Company* (USA) which resulted in a granted patent (US7966909B) [H] for a new HIPIMS-based process for manufacturing razor blades with high aspect ratio cutting edge and, therefore, improved shaving properties. The patent is the property of Gillette with Ehiasarian and Hovsepian listed as named inventors;
- a 3-year (from 2013) research and development contract with *Rolls Royce* to develop HIPIMS technologies appropriate to aero-engine turbine blades [J].

Economic impact through discipline leading activities

The symbiotic joint SHU-Fraunhofer HIPIMS Research Centre provides technical and infrastructural benefits to both partners. The resultant "critical mass" of HIPIMS expertise encompasses the capability needed both to deliver large projects and also to support strategic industrial development programmes in the UK, Germany and worldwide. This has resulted in a large EPSRC-supported collaboration on high efficiency solar-cell glass panels [viii] with *Pilkingtons* (UK), *Gencoa* (UK) and *Von Ardenne* (Germany). The Joint Centre has also started to deliver HIPIMS expertise to other companies such as *Bosch* and *DOT GmbH*, Germany [B]. Since the prime function of the **Fraunhofer IST** is KT and technology transfer, **it can be viewed as a beneficiary** under REF impact, as can the **collaborating industrial partners**.

A further broadening of the MERI HIPIMS group's impact relates to the discipline-leading activities through which it has expanded the field of HIPIMS and, in particular, its industrial uptake. This expansion has stimulated the development of a network of sub-system and component providers who are a **final group of beneficiaries**. This includes providers of analytical equipment, specialist power supplies and magnetron systems and targets. This network is demonstrated by, for example the ~20 industrial exhibitors at the 2012 and 2013 annual International Conferences on HIPIMS [K] run by MERI and the Fraunhofer IST.

5. Sources to corroborate the impact

[A] Patent EP1260603B1 http://www.google.com/patents/EP1260603B1?cl=en
[B] The **Joint Sheffield Hallam University-Fraunhofer HIPIMS Research Centre** is noted on http://hipims.fraunhofer.de/en/collaboration/ and can be corroborated by the Director of Fraunhofer IST, corroborating source 1
[C] Chief Technical Officer Ionbond AG, corroborating source 2
[D] CEO Hauzer Techno Coating AG, corroborating source 3
[E] Patent GB0607269B CN101461032B https://www.google.com/patents/CN101461032B?cl=en
[F] CEO Huettinger Electronik Sp. z.o.o., corroborating source 4
[G] Quotes from representative of, Space Science Technology Department, Rutherford Appleton Laboratory, UK in relation to HIPIMS coatings developed for satellite cryo-coolers http://rseeccleston.blogspot.co.uk/2013/01/meri-pvd-coatings-heading-for-space.html
[H] Patent US7966909B www.google.co.uk/patents/US7966909
[J] Head of Surface Engineering, Rolls Royce, corroborating source 5
[K] International Conference on Fundamentals and Industrial Applications of HIPIMS industrial exhibitors lists 2012 http://extra.shu.ac.uk/hipimsconference/program.php (bottom of page) and 2013 http://www.hipimsconference.com/