UNIVERSITI PUTRA MALAYSIA

PARAMETRIC ANALYSES FOR A SINGLE LAYERED AEROGELBASED COATING USING THERMAL SPRAY FOR AEROSPACE APPLICATIONS

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By

MUHAMMAD IBRAHIM-NADIIR BHEEKHUN

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirement for the Degree of Doctor of Philosophy

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DEDICATION

"Praise be to Allah, Who hath created the heavens and the earth, and hath appointed darkness and light".

[Al-An'am: 1]

"Lo! Allah and His angels shower blessings on the Prophet. O ye who believe! Ask blessings on him and salute him with a worthy salutation".

[Al-Ahzab: 56]

To my Parents

&

To the Love of my Life

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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Chair : Abd Rahim Abu Talib, PhD, PEng, PTech
Faculty : Engineering

Since its discovery in 1931, silica aerogel has been incorporated mainly as flexible blankets for thermal insulation applications in the aerospace arena. However, despite being considered as the world's best thermal insulator it cannot be often applied due to weight and space constraints. This research proposes an innovative route to achieve a single-layered micro-thick thermally sprayed coating and its targeted objectives are ranking the available aerogel powders for plasma spraying based on their evaluated physical properties, formulating and analysing the parameters of an optimised spray-dried aerogel-based powder for atmospheric plasma spraying (APS) and deducing the spraying conditions for surface adhesion of a single layered aerogel-based plasma sprayed coating. Firstly, the different silica aerogel powders were characterised to value their physical properties and their suitability for atmospheric plasma spraying (APS) and suspension plasma spraying (SPS) were ranked. Secondly, slurries of ceramics composed of aerogel and yttria stabilized zirconia were formulated followed by spray-drying, subjected to the Taguchi Design to obtain an optimised sprayable granulated powder. Technical installations and plasma spraying design parameters such as nozzle diameter, electric power, plasma gas flow rate, carrier gas flow rate, concentration of aerogel and dispersant were varied methodically until it could be deduced whether an adhering coating could be achieved or not using the selected aerogels. Characterisation of the coatings using scanning electron microscopy was then performed. Out of six aerogel powders, only GEA™ 0.125 and Enova® IC3100 were opted for plasma spraying. After statistical analysis, the optimised powder YSZ-aerogel spray-dried powder was characterised and had a median particle size of 28.932±0.726 µm, volume fraction of 64.450±0.535 vol.% and uniformity of

0.475±0.002μm. An amelioration in the morphology of the aerogel particles from irregular shapes to spherical and donut-like granulated YSZ-aerogel particles was observed. However, due to low yield of only 10%, the granulated powder could not be opted for subsequent APS. A coating was achieved using APS of as-received GEA™ 0.125 under spraying conditions of a nozzle diameter of 4 mm powered at 25 kW with a plasma gas rate of 45+5 slpm and carrier gas flow rate of 8.1 slpm. The coating had a bimodal microstructure with a thickness ranging from 77.9 μm to 132.0 μm. SPS was not successful neither with GEA™ 0.125 nor Enova® IC3100. Blockage due to agglomeration prevented the injection of GEA™ 0.125 into the plasma. Enova® IC3100 could nevertheless be propelled onto the glass substrates but no melting of particles occurred which was thought because of insufficient power supplied by the plasma gun. Conclusively, not all available aerogel powders can be used for thermal spray applications. Tailoring aerogel particles using spray-drying to make them sprayable using APS is not necessary as it had been proven that GEA™ 0.125 can be deposited in its as-received powder form using APS. SPS seems to be promising using Enova® IC3100 and a suspension-plasma sprayed coating can be achieved using a plasma gun with an electric power higher than 40 kW.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

ANALISIS PARAMETRIC BAGI SALUTAN BERASASKAN AEROGEL BERLAPISAN TUNGGAL MENGGUNAKAN SEMBURAN HABA UNTUK PENGGUNAAN AEROANGKASA

Oleh

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Semenjak penemuannya pada tahun 1931, aerogel silica sudah sebati sebagai satu-satunya salut fleksibel bagi penggunaan penebat haba di dalam arena aeroangkasa. Walaubagaimanapun, meskipun dianggap sebagai penebat haba terbaik di dunia, aerogel silica tidak dapat digunakan secara lazim disebabkan oleh kekangan berat dan ruang. Kajian ini mencadangkan suatu kaedah inovatif untuk memperoleh satu salutan semburan berhaba berlapis tunggal berketebalan mikro dan objekti sasaran inovasi ini ialah melakukan pemeringkatan bagi beberapa serbuk aerogel sedia ada untuk semburan plasma berdasarkan penilaian sifat fizikal mereka, memformulasi dan menganalisis parameter serbuk yang telah dioptimumkan bagi semburan plasma astmosfera (APS) dan yang terakhir meramalkan keadaan semburan bagi lekatan permukaan untuk salutan yang disembur dengan plasma berasaskan aerogel lapisan tunggal. Pertama, beberapa serbuk aerogel silica yang berlainan telah dicirikan untuk menilai sifat fizikal mereka, dan dengan itu kesesuaian mereka untuk semburan plasma atmosfera (APS) dan semburan plasma suspensi dapat dikenalpasti melalui kaedah pemeringkatan. Kedua, buburan seramik yang terdiri daripada aerogel dan yttria stabil telah diformulasikan diikuti dengan semburan kering, dengan merujuk reka bentuk Taguchi untuk mendapatkan butiran serbuk optimum yang boleh disembur. Pemasangan teknikal dan parameter reka bentuk semburan plasma seperti diameter muncung, kuasa elektrik, kadar aliran gas plasma, kadar aliran gas pengangkut, kepekatan aerogel dan bahan penyebar telah diubah secara teratur sehingga dapat disimpulkan sama ada salutan yang melekat boleh dicapai atau tidak dengan menggunakan aerogel terpilih. Pencirian salutan tersebut yang menggunakan imbasan mikroskopi elektron telah dijalankan kemudian. Daripada enam serbuk aerogel, hanya

GEA™ 0.125 dan Enova® IC3100 dipilih untuk penyemburan plasma. Selepas analisis statistik, serbuk semburan kering aerogel YSZ yang telah dioptimumkan telah dicirikan dan mempunyai saiz median zarah 28.932±0.726 µm, pecahan isipadu sebanyak 64.450±0.535 vol.% dan keseragaman sebanyak 0.475±0.002µm. Suatu pembaikan di dalam struktur zarah aerogel daripada bentuk yang tidak seragam kepada bentuk sfera dan butiran zarah YSZ aerogel yang kelihatan seperti donut telah diperhatikan. Walau bagaimanapun, disebabkan oleh hasil keputusan yang rendah iaitu hanya 10%, butiran serbuk tersebut tidak boleh dipilih untuk APS berikutnya. Satu salutan telah berjaya dibentuk dengan menggunakan APS GEA™ 0.125 seperti yang sedia ada di bawah keadaan semburan dengan diameter muncung selebar 4mm dengan kuasa elektrik 25 kW serta kadar aliran gas pengangkut sebanyak 8.1 slpm. Salutan ini mempunyai mikrostruktur dwi-mod dengan ketebalan di antara 77.9 µm dan 132.0 µm. SPS tidak berjaya sama ada dengan GEA™ 0.125 atau Enova® IC3100. Sumbatan yang disebabkan oleh aglomerasi menghalang suntikan GEA™ 0,125 ke dalam plasma. Namun demikian, Enova® IC3100 berjaya digerakkan ke atas substrat kaca tetapi tiada peleburan zarah yang berlaku, difikirkan bahawa keadaan ini disebabkan oleh kuasa yang dibekalkan oleh pistol plasma tidak mencukupi. Kesimpulannya, tidak semua serbuk aerogel yang sedia ada boleh digunakan dalam kaedah semburan haba. Penyesuaian zarah aerogel dengan menggunakan semburan kering agar bahan itu dapat disemburkan menggunakan APS adalah tidak perlu kerana telah dibuktikan bahawa GEA™ 0.125 boleh didepositkan ke dalam bentuk serbuk seperti yang sedia ada menggunakan APS. SPS seolah-olah lebih berkesan dengan menggunakan Enova® IC3100 dan satu salutan semburan plasma suspensi boleh dihasilkan dengan menggunakan pistol plasma yang mempunyai bekalan elektrik lebih daripada 40kW.

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I certify that a Thesis Examination Committee has met on 18 April 2017 to conduct the final examination of Muhammad Ibrahim-Nadiir Bheekhun on his thesis entitled "Parametric Analyses for a Single Layered Aerogel-Based Coating using Thermal Spray for Aerospace Applications" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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LIST OF ABBREVIATIONS

AMREC	Advanced Materials Research Centre
APS	Atmospheric Plasma Spraying
BET	Brunauer–Emmett–Teller
D_{50}	Median Particle Size
CGDS	Cold-Gas-Dynamic Spraying
GEA	Green Earth Aerogels
GEAT	Green Earth Aerogels Technologies
HPPD	Hypersonic Plasma Particle Deposition
HTSCD	High Temperature Supercritical Drying
HVOF	High-Velocity-Oxy-Fuel
HVSFS	High Velocity Suspension Flame Spray
IUPAC	International Union of Pure and Applied Chemistry
LTSCD	Low Temperature Supercritical Drying
PDI	Polydispersity Index
PEG	Polyethylene Glycol
PVA	Polyvinyl Alcohol
RHA	Rice Husk Ash
RSCE	Rapid Supercritical Extraction
SEM	Scanning Electron Microscopy
SLPM	Standard Litre per Minute
SPCTS	Science of Ceramic Processing and Surface Treatments
SPPS	Solution Precursor Plasma Spray
SPS	Suspension Plasma Spraying
SSA	Specific Surface Area

TC4	Titanium Alloy Ti-6Al-4V
TEM	Transmission Electron Microscopy
TEOS	Tetraethyl Orthosilicate
TMOS	Tetramethyl Orthosilicate
TP-CVD	Thermal Plasma Chemical Vapour Deposition
TPFE	Thermal Plasma Flush Evaporation
TP-SP	Thermal Plasma Spray Pyrolysis
UPM	Universiti Putra Malaysia
US	United States
USD	United States Dollar
YSZ	8% Yttria Stabilised Zirconia

CHAPTER 1

INTRODUCTION

1.1. Research Background

With the continuous rise in energy consumption over the globe, proliferating energy charges due to the limited supply of fossil fuels plus global warming and climate issues, both governments and industry sectors supported by the academia are pursuing for alternative or improved thermal systems by employing high performance insulative materials. In aeronautics, the quest for ever more efficient aero-propulsion systems has been constantly motivated by perpetual developments in a wide range of fields including turbine design, combustion analysis, heat management and material sciences. The key drivers behind these evolutions are the demands for reduced weight and increased fuel efficiency while simultaneously reducing the emissions and noise levels (Finley, 2008).

On the other hand, silica aerogel, invented in 1931 by Samuel Kistler, is an illustrious nanostructured material considered as the world's paramount thermal insulator and second lightest solid after being surpassed by graphene aerogel in 2013 (Worsley et al., 2014). By definition, an aerogel is a gel in which the liquid phase has been replaced by a gas in such a way that the solid network is being retained with only a slight or no shrinkage in the gel (Kistler, 1931). In consequence, an open-cell network consisting of nano-sized pores is developed thereby unfolding further numerous exceptional characteristics. Within the class of aerogels, silica aerogel which is the porous nanostructured form of silica dioxide, exhibits the most fascinating properties such as low thermal conductivity (~0.012 W/mK), ultralow bulk density (~0.003 g/cm^3), optical transparency in the visible spectrum (~99 %), high specific surface area (~1000 m^2/g), low dielectric constant (~1.0-2.0), low refractive index (~1.05), low sound velocity (100 m/s). Due to its attractiveness and being the first aerogel invented, silica aerogel is often referred as simply aerogel. This work consequently adopts this shortened term as well.

Since early ages, fundamental studies on aerogels have been dwelled behind significantly in the academia and hence industries due to its high manufacturing cost. This further hampers its feasibility for integration into the commercial market. However, considerable enhancement started taking place at the dawn of this millennium. Numerous scientific reports have been published since then showing not only the enthusiasm but also the scientific understanding of these nanostructures. Nowadays, one of the most focusing areas is the modelling of the thermal behaviour of granular- and fibre-based silica aerogel and its composites (Bi, Tang & Hu, 2014; He & Xie, 2015; Hostler, Abramson, Gawryla, Bandi & Schiraldi, 2009; Wei, Liu, Du & Zhang, 2012; Wei, Liu,Zhang, Yu & Du, 2011; Zhao, Duan, Wang & Wang, 2012a; Zhao, Duan, Wang & Wang, 2012b). On the market level, large scale production of the material has been achieved by various producers such as the North American-based industrials Cabot Corporation® and Aspen Aerogels® and Nano Hi-Tech in China which is considered to be the third player followed by Korean JIOS amongst others. In contrast to these aforementioned companies which produce synthetic-based aerogels using usually tetraethyl orthosilicate (TEOS) as the precursor of silica, there are also other manufacturers which synthesise ecological aerogels from rice husk ash such as the Malaysian-established Maerotech SDN BHD and the Spanish Green Earth Aerogel Technologies (GEAT). These biologically-derived aerogels have both potential cost and environmental benefits. It is noteworthy to mention also that silica aerogels can be prepared in different forms such as in monolith, granules and fine powder. As a result, aerogels are often required to be tailored specifically to develop innovative solutions to today's problems. Their applications are considered to be almost illimitable, tracing their way in different branches such as thermal and acoustical insulation, kinetic energy absorption, electronics, optics, chemistry and biomedical amongst others.

As far as aerospace is concerned, aerogels have been used extensively by NASA since 1992 for astronautical missions in the form of hypervelocity particle capture and thermal insulator, both in the shape of blocks. Recently, Aspen Aerogel® has brought forward aerogel-based flexible blankets for high temperature use up to 650°C for both thermal and fire protection in aerospace systems. However, due to the constraint of weight and space saving, blankets are not encouraged by aero-engine manufacturers. Instead, micrometre-thick thermally sprayed coatings are opted for protection against high temperature, wear and corrosion such as thermal barrier coatings (TBCs) in gas turbines (Kumar & Kandasubramanian, 2016). Longo (1992) reported that some gas turbine engines have nearly 5500 parts which are thermally sprayed to enhance their performance, reliability and durability. Thermal spray processes are extensively used because virtually all materials including metals, superalloys and ceramics can be deposited but it is considerably challenging to retain the characteristics of the nanostructured materials during deposition, which in turn gives rise to a series of research.

Till date, no investigation has been carried out and as a result, the feasibility to achieve a single layered aerogel-based thermally sprayed coating is being established. A single layered thermally sprayed coating is regarded as one which consists of either only type of particles or an agglomeration of different ceramics particles which is deposited directly onto a substrate. The coating system does not comprise of any bond coat nor is part of a multi layered or graded coating. While this work concentrates on achieving a single layered aerogel-based adhering coating, it also targets the coating to consist a bimodal microstructure. A bimodal microstructure is one which includes molten particles acting as a binder which upholds the coating integrity while the semi-molten ones contains nanostructures residing in them (Lima & Marple, 2007). This is attained by setting the operational conditions in such a way that part of the particles is completely melted and some only partially. Such coating microstructure is intrinsically related to thermal spraying of delicate nanostructured materials (Pawlowski, 2008), thus making aerogel eligible to be taken into consideration for thermal spraying, whether by conventional or suspension approach.

This research hence intends to develop a single layered aerogel-based thermally sprayed coating by firstly ascertaining the suitable aerogel powders for the application of plasma spraying, a versatile type of the thermal spray technology. The study further considers the possibility of obtaining an optimized agglomerated spray-dried aerogel-based powder as feedstock to allow or improve deposition. The practicability of spraying aerogel powders using the conventional atmospheric plasma spraying (APS) and the advanced suspension plasma spraying (SPS) in order to achieve an adhering coating on glass substrate is then carried out after which a microstructural analysis of the resulting aerogel-based plasma sprayed coatings is performed.

1.2. Research Gap

Aerogels are considered to be established in terms of their manufacturing process thereby having now an array of synthesis methods which have overcome, to a considerable extent, the high manufacturing cost arising from the source of silica or drying method. The current trend is now inclining towards the development of innovative solutions by tailoring their properties or synthesising composites of theirs to specifically address practical problems. Beneath the aerospace dome, applications of aerogel are still emerging and amongst are thermal insulation systems which include mainly aerogel-doped flexible blankets operating from cryogenic to extreme temperatures for astronautical applications. These are typically composites of silica aerogel particles dispersed in a reinforcing fibre matrix that turns the brittle aerogel into a durable and flexible insulating mat (Fidalgo, Farinha, Martinho & Ilharco, 2013). A study was conducted to evaluate the sustainability of these blankets during hydrogen-fuelled flights at hypersonic speeds ranging from Mach 5 to Mach 9 in harsh thermal environments (Sharifzadeh, Verstraete & Hendrick, 2015). Fesmire (2016) tackled a common problem to both space launch applications and cryogenic propulsion test facilities of providing suitable thermal insulation for complex cryogenic piping, tanks, and components that cannot be vacuum-jacketed or otherwise be broad-area-covered by the use of a layered composite insulation system made of aerogel blankets. A benzoxazine organic-inorganic hybrid aerogel blanket was recently developed in a one-pot sol-gel synthesis to obtain an easy to handle, light, superinsulating material for space applications (Berthon-Fabry, Hildenbrand & Ilbizian, 2016). There has been only one feasibility study that has been carried out on aerogel for aeronautical integration wherein the developed product is again an aerogel-based blanket as well. The work, accomplished by Aspen Aerogel® consisted of evaluating the flammability of the blanket to protect components such as pipes, wires, electronic accessories within the fire zones of aero-engines. The fact of being an inorganic and inflammable material with a constant operating temperature ranging from -273°C to 650°C and a high melting point of 1400°C upholds silica aerogel an excellent firewall. The investigation consisted of a thickness of 7 mm mat subjected to a flame at a temperature 1100°C for at least 15 minutes. The temperature on the rear side of the wrapped system did not exceed 150°C. The product, Pyrogel® 6350 has yet to be integrated for aeronautical applications. The thermal loss that happens when the blankets are under constant vibration and gravitational stress through repeated thermal cycles is still under study.

In the quest for renewable energy systems, substantial efforts are being made towards green architecture and consequently aerogels are tracing their way into building insulation at a faster rate than any other applications. Existing solutions comprise of aerogel-based blankets, powdered aerogel-doped paints, translucent aerogel pellets and aerogel fillers in vacuum packed insulation sheets (Abdul Mujeebu, Ashraf & Alsuwayigh, 2016; Baetens et al., 2010; Koebel, Rigacci & Achard, 2012; Riffat & Qiu, 2013). A mortar-applied coating which consisted of a mineral and/or organic hydraulic binder, an insulating filler comprising of hydrophobic silica aerogel, a structuralizing and additives was developed for thermal insulation of buildings' multi-layer exterior wall structures (Ibrahim, Biwole, Wurtz & Achard, 2014). Furthermore, a research was conducted to develop an ultra-lightweight cementitious composite having both excellent mechanical and thermal insulating properties (Hanif, Diao, Lu, Fan & Li, 2016). It is worthwhile to mention that along with the above expansions and applications, the oil and gas industry remains the undisputed sector which benefits these aerogel technologies to the largest extent due to their compatibility of having superior thermal performance and improved chemical/pressure resistance and large-scale usage coupled with assembly cost savings and ease of use.

Motivated by global CO_2 emission and space saving restrictions, this research proposes a novel route to implement aerogel in aeronautics as a micro-thick single layered thermally sprayed coating. It can be anticipated that limited related studies have been conducted due to the restrictions that exist in aerogel powders such as irregular particle shape, inadequate particle size distribution, ultralow density, possibility of the nanostructures to be annealed in plasma and unknown adhering ability. A study was carried out by Zulkifli, Yajid, Hamdan and Muhid (2014) to reconstitute aerogel particles, more precisely, the Malaysian-made Maerogel (now Hamzel®) using a secondary ceramic, soda-lime, via spray-drying but the study did not highlight whether the process can be optimised for thermal spraying. The lack of understanding of the effect of the formulation and granulation variables on the granulometric properties and morphology of the reconstituted spray-dried aerogel is still present. It is also not confirmed whether spray-drying aerogel is a repeatable process and whether the process is economically feasible, in other words, whether or not the yield of the aerogel-based spray-dried powder is sufficient enough to carry out APS in an efficient manner. Although that a patent (Newman & Lauten, 2008) was filed on applying the thermal spray technology on aerogel to coat a missile surface that would act as an insulation layer, aerogel was not thermally sprayed directly onto the substrate (steel) but instead there was an alloy bond coat (NiCrAlY) in between the substrate and the upcoming aerogel layer, which means that the coating system was not a single layered one. In addition, the silica aerogel being deposited was not on its own but rather as agglomerated ceramic particles. The patent (Newman & Lauten, 2008) employed plasma spraying which is a technique that can literally deposit any powder onto any substrate but retaining the microstructural characteristics of the starting material so that the coating can exhibit comparable properties is challenging. The appropriateness of plasma spraying aerogel particles was not explained and the claims did not prove whether the coating has a skeletal microstructure similar to that the starting aerogel. The patent did not establish whether a critical aspect of bimodal microstructure was achieved. A bimodal microstructure is one which consists of partially molten particles and fully molten particles in order to retain the properties being possessed by the feedstock. Another shortcoming was that coating developed had a thickness of 1.5 to 2.0 mm. The patent was filed nine years ago and till date no further studies were brought forward after it depicting a significant discontinuity of research during which aerogel particles have been modified in terms of their granulometric properties by the manufacturers due to new methods of synthesis. The granulometric properties are considered to be the most critical characteristics to be known when considering thermal spraying technique. Ecological aerogels were not available at that time. Recently, another multi-layer concept was proposed by Jin et al. (Jin et al., 2015) thereby yttria stabilised zirconia (YSZ) and silica aerogel was used in conjunction to improve the thermal insulation capability of an aerospace graded titanium alloy (Ti-6Al-4V or TC4) using the APS technique. However, the aerogel was not thermally sprayed but rather adhere onto the surface of the alloy using an organic glue as a transitional layer or bond coat between the substrate TC4 and the upcoming plasma sprayed YSZ. On the other hand, suspension plasma spraying (SPS) of aerogel has not been reported anywhere so far.

In contrast to the works reported above, this research focuses on achieving a single layered aerogel-based coating for various reasons. The first, being the fact that, no such investigation has been carried out previously. Secondly, multi-layered coating systems such as Thermal Barrier Coatings (TBCs) in aero-engines usually experience delamination in between the two different layers of the coating due to thermal shocks as the coating system consists of different deposited materials which have their own thermal expansion coefficients (Kumar & Kandasubramanian, 2016). Hence, to prevent any delamination in the aerogel-based coating system, if intended to be used as a thermal insulative coating, it is foreseen that a single layered coating would be more suitable. Also, the thickness of a multi layered coating system is often of millimetre order, greater than 1.0 mm rather than of micrometre order.

Overall, it can be argued that there is still no answers whether (1) the existing types of aerogel powders can be considered for thermal spray applications, more precisely APS and SPS (2) as-received aerogel particles can be optimised to facilitate the thermal spraying due to its unfavourable granulometric and morphological features (3) aerogel can be practicably deposited as a single layered coating using the opted thermal spray technology and (4) the morphology of the deposited aerogel-based coating consists of any microstructural characteristics resembled to that of aerogel.

This study uses a systematic approach to find remedy to these technological queries through its targeted objectives in the process of achieving a single layered aerogel-based plasma sprayed coating on glass in order to obtain a clearer microscopy view of the microstructures of the aerogel layer.

1.3. Research Objectives

1.3.1. Aim

To perform parametric analyses for a single layered aerogel-based thermally sprayed coating

1.3.2. Specific Objectives

The specific objectives of the study are as follows:

I. To rank as-received aerogel powders for atmospheric plasma spraying and suspension plasma spraying based on their physical properties

II. To formulate and assess the parameters of an optimised spray-dried aerogel-based powder for atmospheric plasma spraying
III. To deduce the optimum spraying conditions for surface adhesion of a single layered aerogel-based plasma sprayed coating

1.4. Hypothesis

This research assumes that it is practically not possible and considered as challenging to deposit aerogel particles on their own using APS due to their irregular particle shape and inadequate particle size distribution. Next, the reconstitution of aerogel particles via spray-drying can be a potential alternative to thermal spray aerogel particles directly onto a substrate. Then this study considers that SPS can be an alternative to deposit pure aerogel particles with appropriate particle size distribution even though their morphology is not spherical. Finally, it is foreseen that a single layered aerogel-based plasma sprayed coating with a bimodal microstructure can be achieved with proper thermal spraying conditions.

1.5. Significance of Research

Silica aerogel, being the most superior thermal insulator and one of the lightest materials, has been perpetually regarded as potential candidate for future superinsulation systems in aeronautics where weight and space savings are fundamental. Previous studies have addressed particularly on the development and evaluation of aerogel-based flexible blankets for such protection uses or multi-layered coatings. This study brings forward a new concept whereby a single layered aerogel-based micro-thick coating is developed using the thermal spray technology, more precisely, plasma spraying thereby opening panoply of prospects. This investigation principally validates the hypothesis whether aerogel powders can be successfully deposited directly as a single layered coating onto a substrate and provides an insight how to achieve such a validation by the proposed methodologies. The microscopic analysis of the deposited aerogel furthermore brings an understanding on how the particles are affected when propelled into a plasma. Implementation of such an aerogel-based coating into aero-engines depends on the type of substrate. Typically, applications can range from adjusting thermal responses for improved performance and reduced deterioration to protecting polymer matrix composites enabling weight savings for equivalent level of fire protection.

1.6. Scope of Study

The research starts with the philosophy that aerogel has ultralow density and lowest thermal conductivity than any other solids which are two crucial factors when considering thermal insulation systems for aeronautical applications due to load and space constraint. **Figure 1.1** provides a philosophical view of the research. The work targets to achieve a single layered aerogel-based coating using thermal spray, more precisely, atmospheric plasma spraying (APS) and suspension plasma spraying (SPS). It devises a methodical approach to accomplish the coating by:

(a) Characterising the physical properties of all the six commercialised powdered aerogels available on the market and assessing their suitability for atmospheric plasma spraying (APS) and suspension plasma spraying (SPS) based on the evaluated characteristics.

(b) Reconstituting suitable aerogel powders via spray-drying using the Taguchi Design for air plasma spraying. This intermediate step intends to produce spherical aerogel-based powders with an optimum median particle size, volume fraction and uniformity prior to APS. In doing so, the effect of the formulation and granulation parameters, such as the volume of solid content, mass ratio of aerogel : YSZ, amount of binder and dispersant, inlet temperature, atomisation pressure and feeding rate on these three aforementioned responses has been reported statistically.

(c) Conducting a parametric investigation to predict the optimum spraying conditions using the suitable types of aerogel with respect to the APS and SPS. The experiment consists of altering the spraying conditions such as the gun nozzle diameter, power, volumetric rates of plasma gas and carrier gas and concentration of aerogel in an systematic manner to overcome the challenges encountered until an adhering aerogel-based coating is achieved or up to the technological limitations.

This study, however, has certain limitations which need to be pointed out. First of all, limited benchmark literature was available on spray-drying of aerogel particles for reconstitution as well as the application of the thermal spray technique on aerogel, hence making it a feasibility study with numerous design parameters. This study considers the use of only one type of plasma gun for both APS and SPS with a maximum power of 40 kW. The analysis of the thermally sprayed coatings being developed is limited to a microstructural observation using SEM.

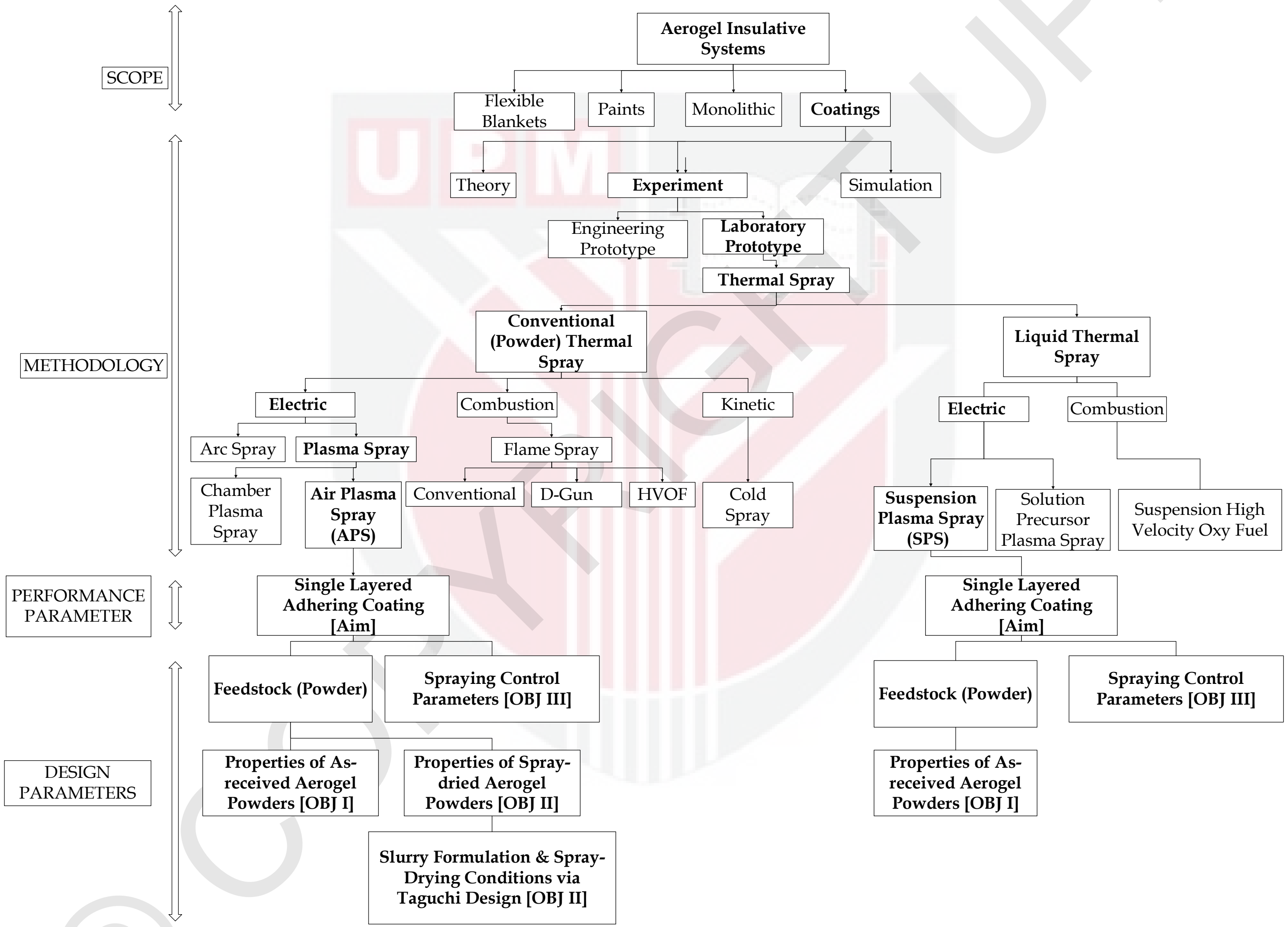

Figure 1.1: Philosophical view of research

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