# Aerosol Sealants Stage 1A: Final Report

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#### **Executive Summary**

A number of test leaks, up to 3 mm in diameter have been remotely sealed

The aerosol is a product of the system parameters, which control

the aerosol **and the individual particles** of the system is designed such that the aerosol particles bond to surfaces when impacting and the individual particles coalesce to form a coherent seal.

The viscous aerosol seal has been the result of:

- a background study on aerosols and sealants;
- a subcontracted project with University of Bristol to develop understanding of aerosol evolution in dry gas atmospheres to produce sealing particles;
- concept development on a number of aerosol generation and conveyance systems;
- a detailed development test programme, culminating in the seal shown in Figure 1.



Figure 1: aerosol Seal on 3 mm Hole

Once produced, the aerosol can be tuned so that it passes through the leak so that it passes through the leak so the leak or deposits on the leak walls . This degree of control suggests that there are options to produce different sealing properties in different sections of the network during an operation, which may provide a route to protect

customer supplies during the sealing operation.

The success of the proof of concept tests indicate that this technology is an ideal candidate for further development to provide the gas industry with an additional tool for use in leak sealing applications in the low pressure network. To achieve this, suggested further work includes:

- Optimisation of the technique
- Alternative chemistries and aerosol formation
- Protecting customer supplies
- Examining differences in natural gas.

It is recommended that this technology is taken to the next project stage.

#### **1** Project Introduction, Changes and Outcomes

This chapter details the aim of the project and challenges met during the project leading to a variation from the original scope of work and the outcomes of the project.

As part of the Stage 1 Seeker Particles work, Steer Energy recognised that a sealant delivered as an aerosol could provide an alternative approach to the existing technologies used to repair leaks. It is appreciated that this is most likely to be directed towards smaller leaks when compared to the robotically applied liquid sealants, and is likely to require some form of enabling technologies to be used to assist that deployment. This work dovetails with the work carried out in Seeker Particles Stage 2 and Gas Polymerisation Stage 1 as well as a number of other intervention projects that SGN are currently running through the NIC / NIA funding mechanism.

Initially this project was to focus on direct technology transfer from the HVAC industry into the Gas industry to produce aerosolised sealants for use in the gas network. Exceptional contractual circumstances prevented this however and a development project was undertaken with a wider overall target.

#### 1.1 Initial Proposal and interaction with University of California, Davis

During the Seeker Particles Stage 1 project, an opportunity for aerosolised leak sealing technology was identified. A proposal was written and accepted by SGN during summer 2014 to transfer aerosol sealants previously developed by Prof Mark Modera for use in the HVAC industry, to the gas industry.

On receiving confirmation of SGN's desire to fund this work, Steer carried out intensive discussions with University of California, Davis, (UCD) over the sub-contract that would allow this work to be carried out within the commercial and legal requirements of the NIA funding provided. During this time, UCD made the decision to licence the patent applications exclusively to a sole entity for all applications to minimise the complexity of their commercial interactions. This was impractical for Steer and certainly outwith the NIA scope of work so discussions were changed to explore the opportunity to obtain a 'carve-out' for the gas industry to allow us to proceed and a timeline for this agreed with Prof Modera. Eventually UCD confirmed that a 'carve out' would not be possible and that we would need to negotiate directly with the future chosen commercial partner. This would most likely considerably delay the start of the project, increase the costs, and it was felt the intellectual property situation would be more complicated to resolve. Following discussions with SGN, it was felt appropriate to look at developing this work with other sub-contractors.

#### **Modera Patents**

An important part of this decision was the need to ensure freedom to operate in respect to existing patents. At the time of the proposal for this work Modera's patents specifically describe their sealant invention as one based on an aerosol of solids. Modera has filed other patents although these focused more on the aerosol generation equipment than fluid dynamics in the conduit. The earliest Modera patents are reaching the end of their enforcement period. The later patents, which will obviously be in force for longer, relate largely to the aerosolisation device. Should these later Modera patents need to be worked around, then the high likelihood that there are suitable alternative aerosolisation techniques available could allow a pipeline technology to be commercialised in under 4 years' time. During the course of the project an additional patent application has been published which is discussed later in this report.

#### 1.2 Project Aim

The original aim of this project was to seal the leaks from the interior of the pipe by releasing aerosolised "sticky" solid particulate sealant materials into the natural-gas distribution system. This was essentially a 'technology transfer' project; taking a technology already applied successfully to the HVAC industry and – with the addition of time responsive sealants – test its applicability in the gas industry.

It is understood that various techniques can be used to produce the aerosol and when properly optimised, these flocculent materials will travel innocuously through the pressurised flow-driven system, and lodge only on the edges of the leaks, and the scope of work was therefore developed to carry out the following:

- Build on the understanding of aerosol transport and leak deposition in ducts.
- Incorporate recent envelope-sealing research on sealant materials that do not remain permanently tacky.
- Refine and utilise CFD modelling techniques to characterise sealant transport, deposition and drying processes.

Building on NIA projects, 'Seeker Particles Stage 2' (NIA\_SGN0050) and 'Gas Polymerisation Stage 1' (NIA\_SGN0058), it was believed that the aim of the project should be widened to "seal the leaks from the interior of the pipe by releasing aerosolised particulate sealant materials into the natural-gas distribution system". This allowed additional sealant options to be investigated.

#### 1.3 Scope of Work

The focus of the project has been extended and the project has changed from a 'technology transfer' project to a more fundamental development project. The Scope of Work "work packages" as described in the accepted proposal and contained within the contract were altered in an updated proposal as follows.

#### WP 1: Application and Sealant Specification

To better understand how the technologies would be deployed in the field, Steer Energy will meet with SGN distribution operators for both Riser and Gas distribution pipelines. This will entail meetings with design and operating engineers, as well site visits in order to characterise the geometry and operating characteristics that are likely to be encountered. This effort will be used to produce a report characterising the distribution of applications likely to be encountered in the Scotia Gas network, as well as a design of a prototype network that can be tested in the laboratory in the next phase.

A review of the Intellectual Property from other areas where aerosols are used will be looked at. This is likely to involve discussions with independent chemistry developers as well as large sealant manufacturers such as Loctite, and Chemence.

Once the initial sealant families have been identified, further understanding will be made through choosing the sealing process that will provide the optimal range of leaks to be addressed through this technology. Key to this will be the sealing process that will be used by the sealants – this could be based on the leak environment (i.e. using environmental factors to act as catalysts, or having to build on existing materials such as hemp / elastomer seals), or could be related to a multiple stage process of delivering an initial chemistry to the seal site, which then is catalysed by a separate chemistry, or by looking at how individual aerosol particles coagulate to form larger sealant volumes.

It is understood that this will be an iterative process and refinements or changes to the chosen chemistries may be made throughout the project.

#### WP 2: Deployment Methodology

Using specialist sub-contractors (i.e. CFP Research and / or University of Bristol) that can provide expertise on the production of aerosols and their transportation, we will seek to answer how variables such as the fluid viscosity, density, sealant components lead to the determining the best dispenser design to produce particles that are driven to the leak site. Part of this will be to understand how different dispenser design, location, orientation, flow rate etc. affects the aerosol particle size and shape, and then how these attributes can be best deployed to optimise the deposition and accumulation of the sealant at the leak site.



Using the sealants that have been chosen and the understanding obtained through the work in looking at the control and optimisation of aerosolisation, transportation and deposition of the sealant, efforts will be made to work out how the transportation and deposition at the leak site can be optimised.

#### WP 3: Design of Aerosol Numerical Model

It is recognised that there are a significant number of physical variables that require to be understood in terms of their impact on the aerosolisation, transportation and deposition of the particles. Rather than carrying out this through experimental means, it is believed that the building of a numerical model using computational fluid dynamics will offer a cost effective way of understanding the impact of each of these components and can then be used to direct the most appropriate experiments.



#### WP 4: Experimental Set up and Testing

A bench top test set up containing scaled model of a riser and a pipe with leaks will be tested in the laboratory. The leaks will be determined as representative and confirmed through discussions with SGN.

Using the sealants and nebulising equipment developed along with the lessons learnt through the work carried out with the aerosol experts and CFD modellers, a set of 'Proof of Concept' experiments will be carried out to determine whether sealing leaks in a low-pressure gas filled pipeline and risers with aerosols has a significant potential for success. Parameters that are likely to be investigated include: particle size, carrier gas flow rate, leak size, shape and orientation, and will investigate the wall and leak deposition and any particular sealing strength.

The level of detail will be discussed with SGN at the appropriate time as there may be more value in focusing more on the numerical model.

#### WP 5: Review of Enabling Technologies

As was highlighted in the work carried out in Seeker Particles Stage 2 and Gas Polymerisation Stage 1, 'enabling' technologies are key to the safe deployment of this type of product. The work carried out in WP 2 - 4 will be important here, and will define the sealing process, and therefore what ancillary technologies are required.

*Computational Fluid Dynamics modelling may also be used to assess the impact of the various techniques on the particle flow within the pipeline system.* 

#### WP 6: Analysis and Reporting

A Steering Committee (made up of representatives from Steer Energy and SGN) will meet on an approximately bi-monthly basis, with input from Technical experts as appropriate during this time. Reports and slide-packs will be produced for each of these meetings.

These reports will detail the analysis that has been carried out in order to determine the appropriate next steps of the project and the specific conclusions that can be made from the design, experimental and numerical modelling work.

#### **1.4 Project Deliverables**

The change in deliverables essentially removed the requirement for the use of a CFD model to be validated by the experimental work. It is appreciated that this would require a significant amount of time and resource to be set up in a non-university laboratory (acknowledging that the CFD model itself needs to be built) and it is viewed as non-essential in terms of the current position of the project and the key questions which are to be addressed. It was believed that the CFD itself could be used to provide indicative results which could then be reviewed and assessed using the experimental work carried out.

The following were target deliverables for the Project:

- 1. Development and application of a test apparatus that can be used to analyse the performance of different sealants, particle sizes, and application protocols. These test results were to be used both to understand the process, and to be documented in the final report.
- 2. Development of a tool for modelling aerosol transport and deposition in pipes and vertical risers. The model was to be documented in the final report.
- 3. The results of a series of 'Proof of Concept' tests and analyses that can be used determine whether sealing leaks in a low-pressure gas filled pipeline and risers with aerosols has a significant potential for success. The final report was to include these results, an explanation of the analyses, and a clear set of recommendations.
- 4. Review of application techniques and modifications that can be made to optimise the delivery of aerosol sealants to the leak site. This work was to include the identification of enabling technologies and products that are required for the safe delivery of the product.

The project also involved a review of Intellectual Property that exists around Aerosol sealants with particular reference to their application within the distribution gas context.

#### 1.5 Change in position during project

The sale of the IP held by University of California Davis has now completed paving the way for collaborative work should this be desirable. During this time further work has been carried out by the team in California with a view to adapting their technology for use in the gas industry. The work presented in this project is complementary to that work so collaboration with that team is attractive and would be beneficial to both sides.

#### **1.6 Project Outcomes**

This project has successfully demonstrated the design and implementation of sealing system

The modified work packages have been completed which has resulted in the following outcomes:

- A study of aerosol types, production and application to the low pressure gas network, in particular riser systems.
- A study of risers with site visits in London and Glasgow to inform understanding of the application of aerosol sealants to the low pressure gas distribution network.
- A simple CFD model providing a first order examination of aerosol particles in a pipe system including leaks.
- A fundamental study of aerosol design and behaviour in the low humidity systems expected in the gas network leading to detailed understanding and control aerosol.
- An experimental programme

seal leaks up to 3 mm in diameters at pressures up to 50 mbar without failure.

It is recommended that this concept technology is taken forward for further investigation and engineering with a view to developing a field ready aerosol based sealant for the low pressure gas distribution network.

It is also recommended that should there be a wish to 'fast-track' the development, discussions are held by Steer Energy with Modera (UCD) and Aeroseal Inc. in order to access their existing IP and jointly develop foreground and operational experience with the technology.

#### 2 Aerosol Sealing in the Gas Network

This chapter provides an introduction to aerosols and a review of IP with a relevance to leak sealing in the gas network. It also contains a review of relevant IP.

#### 2.1 What is an aerosol?

The definition of an aerosol is open to some interpretation. Wikipedia describes Aerosols as <sup>1</sup>:

"An aerosol is a colloid of fine solid particles or liquid droplets, in air or another gas. Aerosols can be natural or not. Examples of natural aerosols are fog, forest exudates and geyser steam. Examples of artificial aerosols are haze, dust, particulate air pollutants and smoke. The liquid or solid particles have diameter mostly smaller than 1  $\mu$ m or so; larger particles with a significant settling speed make the mixture a suspension, but the distinction is not clear-cut. ... technological applications of aerosols include dispersal of pesticides, medical treatment of respiratory illnesses, and combustion technology. Diseases can also spread by means of small droplets in the breath, also called aerosols."

Terms used to describe aerosols are, like their distinction from a suspension or a vapour, not always clear cut. With this caveat, the following graphic by Washington University in St Louis is an excellent overview of the scales associated with common terms and familiar aerosols.



Particle Diameter (µm)

Figure 2: Size ranges and terminology related to aerosols<sup>2</sup>

<sup>&</sup>lt;sup>1</sup> http://en.wikipedia.org/wiki/Aerosol

<sup>&</sup>lt;sup>2</sup> http://www.aerosols.wustl.edu/education/AerosolBasics/Size%20range.htm

#### 2.2 Using Aerosols to Seal Gas Leaks

The existence of Aeroseal and the fact that their patented technology has been used in the US for over 10 years to seal HVAC ducts demonstrates that aerosols can be used to seal gaseous leaks. To further understand how this is achieved it is important to understand the behaviours of aerosols in gas streams.

#### **Aerosol Fundamentals**

An aerosol is a distribution of particles suspended in a gas medium. The length of time of particle remains in suspension is heavily dependent upon the size of the particles and the environmental conditions of that gas. Particles will tend to settle out due to gravity however certain factors will counteract the gravitational effect such as collisions between gas molecules and the particles. Brownian motion sees an individual aerosol particle impacted by individual gas molecules. Gas flow sees an individual aerosol particle impacted by the drag forces of many gas molecules acting in unison to carry the particle with the gas.



#### Figure 3: **Brownian motion**, a large aerosolised particle is caused to move by the collision of many far lighter and far faster moving gas molecules around it. The path it takes is mathematically described, by a 'random walk'

The limit on the size of an aerosol particle is a result of how the ratio of the particle's volume to its cross sectional and surface area varies with scale. The forces transferred by Brownian motion increase with increasing surface area, but the resistance to motion and the settling force of gravity increase more greatly with mass which is proportional to volume. This is most readily seen for a solid sphere, where the ratio of the volume (and therefore mass) to the surface area increases in proportion to its radius.



*Volume* =  $\frac{4\pi}{3}r^3$ ; *Cross Section*  $= \pi r^2$ ; *Surface Area*  $= 4\pi r^2$ 

#### Figure 4: The volume and areas of a sphere

This can be translated as the suspension time for the particles; the key figure in understanding the particle behaviour is the terminal velocity, or settling velocity due to gravity. This is given in Figure 5 which shows the speed at which particles drop out of suspension quickly becomes significant as particle diameter increases.

Size of fog particle	Setting Velocity due to gravity (millimetres per hour)	
[diameter in microns / µm]		
0.2	8.14	
1	125	
2	468	
3	1,026	
4	1,800	
5	2,794	
6	3,996 [4 metres]	
8	7,056 [7.05 metres]	
10	11,016 [11 metres]	
20	43,560 [43.5 metres]	
30	97,920 [97.9 metres]	

#### Figure 5: Settling Velocity for Water Droplets of Different Sizes

In a pipeline system the flow path of the gas is critical to maintaining suspension of the particles. A rule of thumb is often proposed to ensure that the particles follow the gas flow stream is that the Stokes number<sup>3</sup> for the flow should be greater than 0.1. Modera, in his Patent US5980984, shows for the case of a particle (p) in a duct (d) that the Stokes number can be determined and then equated as:

Stokes number 
$$= \frac{\rho_p d_p^2}{18\mu_q} \frac{u_d}{d_d} \le 0.1$$

- $\mu_g$  kinematic viscosity of the gas
- $\rho_p$  density of the particle
- *d*<sub>d</sub> *diameter of the duct*
- *d*<sub>p</sub> *diameter of the particle*
- *u<sub>d</sub>* speed of the flow far from the particle

Where particle density is taken, as an order of magnitude, to be that of water; and dynamic gas viscosity is taken, as order of magnitude, to be that of methane at 1 bar, then this reduces to:

$$\frac{d_{d,mm}^{3}}{d_{p,\mu m}^{2} f_{m^{3}/hr}} \ge 2000$$

*f* Volumetric flowrate along duct N.B. units indicated as second subscripts

In the specific case of gas leaks, and entraining particles from the bulk flow into them, it is advantageous if a large portion of the flow stream is affected by the leak. The dividing suction streamline is the path for gas flow on one side of which gas flow is drawn into the leak, and upon which the other the flow continues along the pipe. Modera offers an expression for this also in his patent. This expression, in a similar way to that for the

<sup>&</sup>lt;sup>3</sup> The Stokes number characterises the behaviour of particles suspended in a fluid flow. The Stokes number is defined as the ratio of the characteristic time of a particle (or droplet) to a characteristic time of the flow.

Stokes number, can be reduced to a simpler form to describe the distance *h* from the pipe wall of the dividing suction streamline in gas risers:

$$h = d_d \sqrt{50.63 \frac{Re_s}{Re_d^{\frac{7}{4}}}}$$

ResReynolds number in the leakRedReynolds number in the duct

This expression, in a similar way to that for the Stokes number, can be reduced to a simpler form to describe the distance h from the pipe wall of the dividing suction streamline in gas risers:

$$h = 0.67 \frac{d_d^{1.75} d_l^{0.5}}{f^{0.875} C_p^{0.25}}$$

*d*<sub>1</sub> The effective diameter of the leak

*C<sub>p</sub>* Mass concentration of the particles in the leak

This expression and that for the Stokes number together suggests some important desirable attributes for aerosol particle sealing. Namely:

- 1. Flow rates should be low, albeit turbulent
- 2. Aerosols will be most effective in the case of larger diameter ducts
- 3. Particles should be input to the gas at a low concentration
- 4. The particles should have a low density
- 5. The particles should be small.

These theoretical attributes have not been proven but provide a useful starting point for the work. Indeed they could, surprisingly, point towards the use of small bubbles being a suitable option. Considering this further, it can be expected that a bubble that can be made lighter than the gas (e.g. with combustible hydrogen, incombustible helium, or a mix thereof) would tend to be drawn into the leak in preference to the denser methane by the flowstream bending towards it. This was the genesis for including the use of bubbles under the umbrella of aerosols for leak sealing.

#### Leak "clogging"

Colloid flows have been studied to understand how the distributed particles interact and can create blockages at constrictions larger than the particle. Typically the work is aimed at better understanding how "clogging" blockages occur and how these can be *avoided*. In the current work it is desired that we form clogs but that same work can be used to understand the clogging we want to encourage.

Clogging occurs not only in colloids but across a range of physical phenomenon<sup>4</sup>, including such different areas as pedestrian flow through a doorway, and sand transport within pipelines, but it has been found these all share the same underlying phenomena. Critically, there is no abrupt onset of clogging but rather a shrinking average time before blocking as critical parameters change. A study specifically into colloid flow<sup>5</sup> in microfluidic passages, very much akin to leaks, found that clogging was *not* effected by particle flowrate or

<sup>&</sup>lt;sup>4</sup> See http://www.nature.com/srep/2014/141204/srep07324/full/srep07324.html

<sup>&</sup>lt;sup>5</sup> Mechanism for clogging of microchannels; Hans M. Wyss, Daniel L. Blair, Jeffrey F. Morris, Howard A. Stone, and David A. Weitz; Phys. Rev. E 74, 061402 – Published 11 December 2006, http://journals.aps.org/pre/abstract/10.1103/PhysRevE.74.061402

volume fraction. Rather, the average time before blocking is seen to scale with the ratio of orifice to particle size.

Whilst this finding might be considered intuitive and not particularly informative, unquantified as it is, the irrelevance of particle flowrate or volume fraction is very illuminating. This provides some confidence that risers will not need to be rapidly flooded with aerosol to be successful in leak sealing. There is therefore an indication of the suitability of carrying out any sealing operation whilst the riser remains in service.

Once a particular aerosol technology has been identified then experiments will have to be carried out to ensure that customer equipment is protected. This may lead to the necessity for 'enabling technologies' to protect against unintentional sealing.

#### **Scenario Traits**

Every leak sealing application is unique and imposes different constraints on what a viable technology can look like. In the case of water in distribution systems, this clearly includes toxicity. In all cases where the approach will be implemented by the public or unqualified technicians, HSE demands must be minimised. In the case of sealing gas distribution systems, including risers, the following unique factors should either be exploited or accommodated:

	Ability to incorporate flammable gases e.g. hydrocarbon propellant	
Network Factors to Exploit	Ability to incorporate clean burning liquids e.g. sealant monomers	
	Gas relative humidity levels that are controlled and low during normal service	
	Requirement to withstand only low pressure differentials in risers i.e. less than 22 mbar	
	In-service maximum flowrates that could be readily and significantly exceeded during and only during the sealing work	
	Very high ratio of pipe diameter to outlet (i.e. nozzle and leak) diameter in comparison to many systems e.g. water distribution, HVAC.	
Network Factors to Accommodate	The leak may be in a leg of the network that is not flowing, cannot flow (e.g. blind spur) or is flowing at high rate. There can therefore be a very wide range in the proportion of injected material that passes the leak and at what concentration.	
	The severity of the leak will affect the gas in the piping: a severe leak will likely lead to the line being isolated and purged before sealing (i.e. non-hydrocarbon working atmosphere) whereas minor leaks may allow the system to remain in service for sealing (i.e. hydrocarbon working atmosphere)	

#### Figure 6: Network Factors

#### 2.3 Prior Art

The key items of prior art are those relating to Prof. Mark Modera's work on aerosol leak sealing. In the first instance this corresponds to a 1996 patent which is due to expire in 2016 (2019 in the US) and therefore is unlikely to impinge upon the work carried out here. A further patent published whilst this project has been carried out is much more pertinent to this work.

Modera's initial patent (US5980984) provides a comprehensive combination of underlying theory, operational design methodology, practical equipment and field trial data in domestic air duct applications. Other inventors' prior art that is cited (see below) does not exploit the deep level of insight in to the behaviour of aerosols flow that Modera has and thereby suffer from much greater operational burdens and constraints.

The patent claims encompass a wide range of embodiments far beyond those represented by the reported laboratory trials and domestic air duct work. The breadth includes:

#### Header text

- Particles of between 1-100 μm
- Driving fluid flowrates of 100-20,000 cubic metres per hour
- Solid or liquid particles, but arguably not a combination
- Solid or liquid particles, but ones that "substantially retain their shape"
- Sealing efficiencies that vary between extremely low and no stated maximum
- Distinct deposition regimes that target leaks or that provide a uniform duct wall coating

However it is also clear that the operating conditions to which this system is most suited are air ducts because:

- The penetration / deposition trade-off suits the approach to restricted lengths
- The presumption that the conduit is not in-service when sealing is being carried out and that therefore:
  - Outlets and sensitive equipment can be sealed off
  - Flowrates can be manipulated (e.g. by a factor of 2)
  - Gauge pressures can be dramatically manipulated (by a factor of 5)
- The flow conditions rely on a significant leak rate that would likely be intolerable for systems containing other than air (e.g. "minimum span of 3 mm or more ... at least 3 mm").

This US patent expires in 2019 but was seemingly extended from a worldwide filing (WO9614166) of 1996 which would give an expiry date of 2016 for the world (20 years).

#### Reflections

It is was our belief at the start of the project that the initial patented technology of Modera missed both some opportunities and some challenges present in low pressure gas distribution system leak sealing.

#### Scope for fresh art

The most significant gap in the patented domain was the use of multi-phase aerosol particles. This might mean:

- Liquid coated solid particles. These are common in atmospheric aerosols e.g. clouds
- Two part systems a solid aerosol, followed up by a liquid aerosol. This would bear some similarity to a two part adhesive system or a catalysed chemical reaction
- Liquid particles encapsulating gas i.e. a bubble or foam
- Liquid particles encapsulated in a solid (or drier and viscous) 'skin'. Micron scale such particles have already been developed and used in self-healing materials technology.

#### Leak size

The approach requires significant leakage flowrates for efficient entrainment to occur and explicitly states that a leak of at least 3 mm span and minimal flow length (i.e. duct wall thickness only) is required. A leak of such a size in a gas system would have to have been overlooked whilst posing a significant public safety risk to require a sealing repair. It should also be noted that the sealing system must address very small leaks as the sealing process requires many particles to complete the overall seal thus as sealing progresses the hole size becomes progressively smaller until it is completely sealed.

#### Particle size

The patent confirms that leaks with a diameter at least 30 times the size of the particle can be clogged and sealed. If such behaviour were to be seen in a gas system then the particles would need to be at least two orders of magnitude smaller than the throats of burner nozzles or be reliably prevented from reaching them.

The stated particle size range might be an avenue for demarking fresh art i.e. working with aerosols below 1 micron, especially given the anticipated smaller leak sizes of a gas distribution system. There could however be major obstacles to this including the health and environmental concerns with the creation and escape of nano-scale particles.

#### Leak criticality

The patent describes an 80% reduction in leakage rate as attractive performance in air duct systems, where the aim is to reduce HVAC energy wastage. An 80% reduction in leakage is unlikely to prove acceptable in most localised gas system leaks i.e. seals need to be 'almost impermeable'. It could be a significant benefit in the case of large stretches of cast-iron pipe with joints suffering micro-leaks.

#### Ingress risk

The air in the HVAC ducting must be purged of the moisture that it contains for the preferred embodiment described to be most effective. A dry environment is arguably more readily attained in a gas system making the approach, and likely other moisture sensitive chemistries, more practicable.

#### Egress risk

The main risk alluded to in the patent is that of loss of the aerosol material from the air duct via outlets into the home or office leading to property damage. In a gas system the risks are of gas egress or air ingress via the leaks creating asphyxiating or explosive mixtures and a consequential loss of life or property.

#### Fluid contents

The HVAC environment provides an aerobic one and one in which volatile solvent evaporation is favoured (i.e. vapour pressure is favourable). However, in a gas system conditions are anaerobic and the evaporation of some volatile solvents will be opposed by the hydrocarbon atmosphere.

#### Flow regime

The manipulation of the flow regime in response to knowledge of the size of the leak is fundamental to achieving the efficiency and control of deposition that Modera describes. It should therefore be anticipated that operational flow regime of a gas system is unlikely to match the requirements for any specific leak sealing scenario. Therefore an aerosol sealing approach may likely require cessation of operation, moderate pressurisation, or elevated flow (e.g. venting/flaring) to attain the necessary aerosol particle behaviour. It could also benefit from 'reversed' flow where access at the top of the riser is preferable for deployment of the Aerosols.

#### **Related Art**

#### **Duct Sealing**

Modera discusses prior art from 1980s which is consequently expired but places the following broad concepts into the public domain:

Name	Number	Major concepts
Shinno	4,327,132	Lining of a pipe via a mist reliant on high flowrate evaporation drying
Koga	4,454,174	Lining of a pipe via a mist reliant on heating and manipulation of gas pressures
Hyodo	4,768,561	Use of aerosol foam (emulsion or latex based) to seal piping leaks delivered from a pressurised can

#### Figure 7: Additional Duct Sealing Patents

The work that cites Modera includes:

Related work by the University of California refining the apparatus for duct sealing with aerosols (US 7156320 B2, US 7851017 B2)

#### Header text

- Coating of air ducts with aerosol system by point to point flow after characterizing the flow path by testing (US 7498056 B2)
- Sealing of duct leaks by the entrainment of solid elements added to the fluid flow for that purpose (US 7810523 B2, WO 2003093713 A2), commercialised by Brinker.
- Sealing of duct leaks by the entrainment and shear induced fibrillation of solid particles suspended in a liquid (US 8015998 B2), commercialised by Sealtite.
- Sealing of gas pipes by disconnection and a series of flushes with polymer suspension (solids in liquid) and heated air before purging and placing back into service (EP 1515077 A1, WO 2005024286 A1)

Other works identified are most applicable in the gas polymerisation work (e.g. see work of JJ Packo) and liquids sealant work (e.g. US 6180169 B1).

The only other minimally invasive work in the context of pipeline sealing that has been found is the expired art of Price [4,994,307 1991; 5,171,771 1992]. The patents describe using an unspecified fogging delivery system to convey polymerisable monomer through a pipe. Specific application to sealing pipe joints incorporating yarn and perished elastomeric seals is made.

Rosin is a naturally occurring material, made from the secreted sap of specific varieties of tree. It is used in varnishes and in its pure form for preparing the bows of stringed instruments for playing. Rosin is used as a component in many leak sealing technologies, including radiator systems (US2315321, US2293546) and pneumatic (rotating) tyres (CN100526412C, CN1052498A). As this is a naturally occurring, naturally tacky, grindable and combustible solid it is certainly worth assessing for use as a gas system aerosolised sealing material.

There is, quite simply, a colossal amount of prior art in the area of aerosol generators and distributors, these are items which will be used by the project but it is not envisaged that new aerosol generators will be designed in this stage of the project.

#### 2.4 Developments in IP and University of California Intellectual Property

The patent (WO 2015/149023 A1), which was applied for on 27<sup>th</sup> March 2014, details a system for sealing leaks in pipes that comprises of:

- a. forming particles of a sealant, the particles having an outer surface with a tack range that diminishes over time
- b. creating a pressure differential between the interior and exterior of the pipes or ducts
- c. flowing the particles through the interior of leaky pipes or ducts
- d. adhering sealant particles to surfaces adjacent to a leak and to other particles to form a seal
- e. wherein sealant particles that do not form a seal will not adhere to interior surfaces of the pipe or duct or to other particles after a period of time or after a certain distance from the particle injection point.

The patent also makes claims about controlling the tack range by controlling the temperature and humidity within the interior of the pipe, to within 40 - 110 °F, and 65% - 95% RH respectively. It also makes the claim about using particles with a diameter in the range of 0.5 to 30 microns, aerosolised from a sealant which comprises 5% to 70% solids fraction, or to use a sealant plus solvent (such as water, acetone, or p-cholorobenzotrifluride). Finally it states that the system is suitable for a pressure differential across the interior to exterior of 0.1 to 100 mbar.

In effect, this patent application can therefore by characterised as having the following key features:

- Aerosolized Particles (i.e. liquid and or solids) which bind on Leaks
- Formulated to dry effectively
- Identifies leaks through marking

There is a sister patent that is closely aligned with this one that utilises the technology for sealing air envelopes in buildings.

#### **3 WP1: Application and Sealant Specification**

This chapter looks at the chosen application of risers and examines different types of sealant with a view to the development of a sealant specification.

The agreed application for this project is riser systems. Results of the project will therefore be tailored towards this application but they will not be limited to use in risers. Any pertinent technology will be addressed towards any relevant gas industry applications.

#### 3.1 Choice and Examination of Target Pipeline Environment

Riser systems were chosen as the target application for aerosol sealants because:

- This is an area of the network which had not been previously specifically targeted from the Seeker Particles project which led to the aerosol proposal.
- The proposed technology transfer from the HVAC sector was seen as pertinent; HVAC systems are selfcontained and can be treated in isolation. The riser system was seen to an extent as being in a similar status.
- The pre-existing riser fogging system is an aerosol approach, albeit unlocalised and with limited control, which provided some validation that a targeted aerosol could work effectively in risers.

Since the project has expanded from HVAC technology transfer, the ground-up aerosol design element allows the project capabilities to be extended to design the aerosol to enable targeting of specific leak areas. This targeting will allow specific areas of the riser to be treated, such as underground portion of the pipe underneath the footings of buildings.

Test-rigs to simulate key elements of a riser system can be built and operated cost-effectively by Steer and therefore they remained the target application for proof of concept. However, the characterised aerosol at the end of this work was clearly seen as equally suited to other parts of the gas network, and similarly other sealing approaches more appropriate for challenging riser scenarios (e.g. gas polymerisation).

It should therefore be noted that this 'target' environment does not preclude the technologies and concepts developed also being reviewed for their suitability in a subsurface environment.

#### **Riser Site Visits**

Visits to 5 different sites were arranged and undertaken during August 2015; 2 in Scotland and 3 in Southern England. These have contributed to the understanding of the different types of riser procedures and preferred operational activity for the different regions.

### In summary, there is an enormous variability between risers (and laterals) in different area and within a single riser itself including:

- Diameter
- Material
- Jointing techniques
- Protection / encasement (e.g. paint, conduit, concrete, soil)
- Configuration i.e. arrangement of branches, tees, crosses, reducers etc.

#### The leaks encountered are of three predominant types:

- 1. New joint leaks: infrequent and readily resolved by replacement after a failed test. These are identified and handled by SGN site teams.
- 2. Old joint leaks: the most common issue with existing stop-gap and permanent repair options. The leak is identified by customers and located/repaired by SGN call-out teams.

3. **Corrosion leaks:** less common but none the less a normal occurrence with the normal approach being to replace the riser. The leak is identified by customers and (possibly after interim repair) the corroded pipe replaced by an SGN site team.

#### **Existing Repair Techniques**

A number of existing technologies are available for the repair of leaks from outside and from within the pipe and these were discussed during the site visits.

#### **External Repairs**

Externally mounted risers can be accessible with suitable access equipment, and there are a number of approaches available to SGN Service Technicians:

- Temporary repairs can be carried out using mastic or heat shrink sleeves. These are applied externally to the leaking section and then checked regularly by SGN Service Technicians.
- 'Polyform' Polymer Wrap is a two part system which once mixed forms a non-slumping paste which is applied to the outside of the joint before having a clear polythene wrap applied to contain the paste as it cures in place.<sup>6</sup>
- Self-amalgamating tape is also being examined for use in the gas network. These systems are already extensively used in electrical systems and addressing leaking water pipes.
- i-Seal is a 'drill and inject' sealant whereby a leaking thread joint is sealed by drilling into the joint and injecting an anaerobic sealant into the threads.<sup>7</sup>

Each of the existing approaches possesses significant benefits, but none offer a universally preferable method.

#### **Internal Repairs**

Internal repairs have the advantage of not requiring access to the pipe and a number of internally applied repair systems exist:

- 'Serviforge' anaerobic riser fogging: The anaerobic riser fogging technology was identified within the IP
  review carried out as part of the project proposal. Essentially, this uses a low viscosity anaerobic sealant
  and transports it (using an air blower and appropriate filters) throughout the riser as such is likely to suffer
  from lack of control of exactly where the sealant is deposited in the system, and is only suitable for minor
  leaks. The system requires all services to be switched off and the product vented from the top of the riser.
- 'Fill and Drain', this is an older version of the anaerobic fogging system which required the riser to be isolated and then filled with a liquid sealant, before being drained down and recommissioned.

#### Conclusions on the decision to have risers as the target application

During the course of the project, information has been sought on risers and this has been used as a reference for the development work of the project. Applications are not limited to risers though and all avenues will be pursued in the event of a suitable application for any new technology.

Key areas of interest for the rehabilitation of risers would be underneath the footings of buildings where excavation is impossible.

Aerosol techniques are already used in the gas industry in the form of fogging technologies. An aim of this project will be to better understand the aerosol properties of these techniques to improve their effectiveness.

#### 3.2 Development of Sealant Options

This section of the report takes a high level look at aerosol types and categorises them prior to examining which are most likely to be best suited to the chosen applications.

<sup>&</sup>lt;sup>6</sup> http://www.mwpolymers.co.uk/files/Polyform Low Pressure Repair System.pdf

<sup>&</sup>lt;sup>7</sup> http://www.synthotech.com/uploads/Docs/ISeal Product Sheet.2015.pdf

In the basic application we are looking for an aerosol which is deployed into the riser, or pipeline system, is then transported through the system to a leak site and then is deposited at the leak and forms a seal.

The aerosol must therefore be able to be generated inside a pipe system, transported through the pipe system and become entrained and seal the leak. The entrainment and sealing phase require matter of sufficient strength to block and seal the leak over the operating pressure range. It should be recognised that the eventual sealant may well be very different than the original material used to generate the aerosol. An initial classification hierarchy for aerosol sealants has been given in Figure 8.



#### Figure 8: Initial classification hierarchy for the range of aerosol concepts to be considered

A variety of means, including patent searches, academic paper review, meetings with academics and bench top testing have been used to generate the specific concepts and chemistries.

#### **Liquid Droplets**

Liquids are easier to aerosolise than solids and are thus, operationally, more attractive for a field deployed leak sealing technology. Aerosolised liquids are however highly dynamic because of the evaporation, condensation and agglomeration that takes place during transit. These phenomena need to be understood fully and tuned if an aerosol sealant is to be designed with predictable and repeatable capabilities e.g. distance of penetration along flow stream. However these phenomena need not be treated as limitations imposed on liquid aerosols. Elegant liquid aerosols will use these phenomena as a part of the sealing mechanism. These systems are the foundations of the research work carried out by the University of Bristol reported in the next chapter. As solid aerosols can be produced from liquids which dry to solids in the aerosol phase, it is therefore no surprise that liquid aerosols have formed the majority of the work of this project. Fogging systems already use liquid

aerosols however these remain as liquids during their implementation. Such liquids may not be viscous enough to resist internal pressure in the case of leakage.

#### **Viscous Liquid**

A viscous liquid may have sufficient resistance to pressure in a leak to form a seal. This is particularly the case for thixotropic liquids. It is challenging to aerosolise highly viscous liquids but is much easier to produce a viscous liquid by evaporation of an aerosol of lower viscosity. Control of the evaporation process can lead to controlled viscosity of the aerosolised particles. If that viscosity is sufficiently high then the aerosol may be suitable for sealing leaks.

#### Solidifying Liquid

Liquid to glass: some solutions, such as sugar in water, form a non-crystalline solid upon drying in the right conditions. An aerosol of such material, possibly partially dried before entering the gas flow path itself, could be allowed to finally dry at the leak site and agglomerate. As the particles would be spherical their behaviour in the flow stream would be comparatively easy to predict and therefore tune via modelling rather than exhaustive testing programs.

Enhanced consolidation of the seal might be attained by introducing aerosolised 'neat' solvent to soften the seal and allow it to 'bed in' and confirm to the leak path walls.

#### **Composite Aerosols**

It is recognised that an aerosol comprises elements of one material suspended in another material so they are inherently 'two part'. In this section we examine aerosol particles that are themselves made up of two different materials.

#### **Two Part Liquids**

Two part adhesives and sealants are commonplace as they offer a long shelf life as well as a short solidification time that is not reliant on external factors or triggers e.g. heat. Their attraction is as strong, or greater, in aerosol based sealants as in other methods because the leak site is inaccessible and possibly un-located.

A fundamental challenge of a two part aerosol however is the challenge of how sufficient mixing can be assured, including:

- How the two parts are aerosolised separately, probably in sequence, so that they do not mix and react before deposition thus becoming simply a single part sealant.
- How sufficient co-deposition of the aerosol is assured within the ratio limits that will ensure the formation of a sound sealant.
- How the two-parts are to diffuse into one another or be mixed sufficiently, especially if they are deposited as films one on top of another.

It is clear the use of a two-part aerosol sealant will introduce additional difficulties not encountered with a single part system. However a two part aerosol may yield benefits over single part aerosols.

#### Liquid Coated Solid Particle

The high evaporation rate of liquid aerosol droplets is a result of their scale: small objects have a high surface area to volume ratio. The evaporation of a droplet in a low humidity atmosphere after aerosolisation will therefore always be high. The evaporation could be slowed down by composing the droplets of a porous solid soaked in the liquid. The ideal candidate would minimise the solid weight and maximise liquid absorption. A dedicated workscope, assessing the use of hydrogels, is included in the work the University of Bristol will carry out, recognising that this exploits an area of significant expertise.

One such solid could be **Super Absorbent Polymers (SAPs)**<sup>8</sup>: which provide exactly the described properties in the case of water and find application in many products including sanitary products. Such particles could be used for the transport of the liquid with minimised evaporation.



*Figure 9: Super absorbent polymer powder before and subsequently the gel formed after the addition of water. This material used by Imperial College London in research on self-sealing concrete*<sup>9</sup>.

#### Liquid Filled Solid

In this section the sealant is in the form of a liquid but it is prevented from adhering to the pipe wall by the micro-capsule outer shell. On impact with the leak, the shell would be broken to release the liquid. This may need to be facilitated by a non-mechanical trigger system.

One of the most fundamental challenges of remote sealing is the transport of the sealant to the leak site without the materials depositing before or after the leak site. A 'sticky' particle could be likely to adhere equally well to the pipe wall as to the leak path surfaces. A way to avoid this is to encapsulate a liquid inside a solid capsule designed to breach at the leak location and contribute its contents to a seal forming reaction. Key to exploiting this mechanism will be identifying the properties of the leak site which can be used to trigger a breach of the microcapsules. This might be as simple as high shear or as selective as pipe wall material trace components

- Encapsulated catalyseable monomer:
  - Prof White's Research Group of the University of Illinois Urbana Champaign have developed a means to manufacture microcapsules of resin and hardener by agitating emulsion<sup>10</sup>. The speed of agitation controls the size of the microcapsules with average diameters in the range of 10 - 1000µm and a standard deviation of less than 35% of the mean value. They use these to create self-healing plastics in which cracks rupture microcapsules of resin and microcapsules of hardener which run and mix under the influence of capillary action. The restoration of material strength is extremely effective.

<sup>&</sup>lt;sup>8</sup> https://en.wikipedia.org/wiki/Superabsorbent\_polymer

<sup>&</sup>lt;sup>9</sup> http://www3.imperial.ac.uk/concretedurability/researchprojects/selfsealingconcrete

<sup>&</sup>lt;sup>10</sup> Journal of microencapsulation, Nov-Dec 2003, Vol. 20, No. 6, pages 719-730



Figure 10: SEM image of the microcapsules produced by UIUC

- Prof Wass's Research Group of the University of Bristol have developed a microencapsulated monomer and particulate catalyst than can be embedded in fibre reinforced plastics to provide self-healing capability.
- Encapsulated water: Co-called "Dry water" is a "powdered liquid" formed by a water-air emulsion in which tiny water droplets are surrounded by a sandy silica coating which prevents the water droplets from combining and turning back into a bulk liquid. The result is a white powder that looks very similar to table salt. The material was re-discovered by researchers from the University of Hull in 2010<sup>11</sup>, a product of the growing work with nanoparticles. Water carried to the leak site could be used in a range of chemical reactions, including with aminosilane chemistries which are being exploited in another SGN-Steer project (see later description of two part systems).
- Encapsulated hydrocarbons: Dr Wuge Briscoe of the University of Bristol has connections with a company which can create polymer coated hydrocarbon droplets. A high viscosity grease may be enough to seal some leaks and avoid the complexity of curing reactions. Dr Wuge's technical expertise lies in the area of surface chemistry which would be essential to identifying non-mechanical triggers for the rupture of all the mentioned microcapsules.

The challenge for all of these concepts is to have the particle rupture only at the leak site. The particles must be resilient enough to undergo an aerosolisation process prior to being transported through the pipe network and then become entrained at the leak site.

#### **Gas Filled Solid**

The review of Modera's work earlier in this report highlighted how lighter-than-air bubbles are an 'ideal' aerosol particle for leak entrainment. This is because they can be large and influenced readily by flow path, neutrally buoyant and thus non-settling and convey comparatively large quantities of material.

• **Discrete Bubbles:** The 1970s children's toy 'super plastic'<sup>12,13</sup>, which is still available, is an acetone dissolved plastic which can be blown into bubbles. Updated equivalents, offering nominally less hazardous tough bubbles (e.g. US Patent 20080176977), demonstrate the availability of other chemistries any or all of which might be suitable for proof of concept. The expectation is that a bubble would rupture when drawn into a leak and deflate onto/into it. Initial bench top testing has developed large (6mm diameter) helium bubbles that, without artefacts of their crude manufacture, would be neutrally buoyant. After the bubble was burst

<sup>&</sup>lt;sup>11</sup> https://en.wikipedia.org/wiki/Dry\_water

<sup>&</sup>lt;sup>12</sup> http://en.wikipedia.org/wiki/Super\_Elastic\_Bubble\_Plastic

<sup>&</sup>lt;sup>13</sup> https://www.youtube.com/watch?v=gcJE2W65Rhs

and the skin rolled up a soft polymer ball of approximately 0.5mm diameter. Discussions were held with Prof Stride of the University of Oxford to explore the feasibility of this in the Riser context.

#### Gas Filled Liquid (Foams)

Foams are widely used for the transport of solids in the oil and gas sector. Foams can also be used to transport liquids in gas media as the bulk density of the foam is much closer to the gas phase than the liquid phase. The foam will easily travel upwards against gravity in a confined pipeline situation. Liquids of sufficiently high surface tension will not only travel as a foam in a gas medium but they will also transport solids in the same medium. Steer have held discussions with Dr Paul Stevenson of the University of Hull on the use of foams in the gas network and separately commissioned a feasibility study on their use.

NB; Initial work has been carried out on foams which has yielded some interesting and potentially valuable results in both internal pipe coating and leak sealing. This work did not use foam directly as an 'aerosol' but utilised the foam for transport purposes resulting in the investigation moving away from the focus of the aerosol project. Further information on this work will be made available to SGN.

#### **Solid Particles**

Tacky particles that will agglomerate to one another are an apparently simple option which Modera has sought to develop patent protection around. There is however prior art in the broader area of leak sealing that exploits tacky particles in liquid leak sealing methods which could offer a source of novel art in gas sealing applications. Other options for solid particles include large surface area solid particles which will be transported more easily than round ones.

A challenge to any solid aerosols is the generation of the aerosol itself. Powders which tend to agglomerate together will be difficult to separate to form the aerosol in the first place. So although solid aerosols will be considered for completeness they may be very challenging to produce.

• **Rosin and Shellac**: are naturally occurring organic materials which can be ground to a fine powder and remain tacky. There is no chemical reaction involved in their agglomeration and, given the low pressure differential across a gas riser wall, compaction to provide a solid seal that conforms to the leak would be comparatively low.



Figure 11: Example of (left) rosin in its processed and subsequently ground form and (right) shellac processed into readily crushable flakes

• Short chopped fibres: the initial interest in short chopped fibre was prompted by their high aspect ratio which will increase the proportion in a flow that would interact with the flow disturbance near to the pipe wall in the vicinity of a leak. Further reading on the surface treatment of fibres ("sizing") to promote adhesion in fibre reinforced plastics and articles upon the properties of spider silk highlighted other advantageous properties. Fibres can be suspended in a gas flow in part because they have a very large surface area that the gas can impart force upon. This surface area also provides a site for

chemicals to promote adhesion to be added. A range of short chopped fibres have been examined, however the fibre length is in the mm range.

Smoke destabilisation: smoke is an aerosolisation of solid particles generated by (partial) combustion. They are common pollutants which has led to technologies to remove them from gas flows, including electrostatic destabilisation by smoke precipitators<sup>14</sup>. Non-conducting liquid streams generate electrostatic charge when flowing which must, depending upon the material, build-up or discharge when the stream passes through a leak in the pipe. If the electrostatic forces involved are large enough and can be relied upon to behave in a repeatable manner it would provide a means to preferentially deposit aerosol particles in a leak.

#### **Gas Augmentation**

The control of the evaporation rate of water will be directly applicable to other solvents and so will form the basis of a solvent let down approach to sealants.

The early stages within Bristol's work, developing the means to control the evaporation rate of aerosolised water, also provides a framework that the gas polymerisation work can use to augment the gas stream with water to enhance polymerisation. This framework will be passed to The University of Liverpool to exploit in the work of Gas Polymerisation Stage 2.

We are also looking at taking lessons learnt from the gas polymerisation project, recognising that the ideas and chemistries generated may be suited to transpiration via aerosols.

#### 3.3 Options to Pursue

The initial options for pursuit are given in Figure 12.

<sup>&</sup>lt;sup>14</sup> See http://www.explainthatstuff.com/electrostaticsmokeprecipitators.html



Figure 12: Initial Options for Examination

The main and clear option is liquid droplets as the vast majority of aerosols used in industrial contexts are liquids aerosolised into gas streams. Other options are composite particles, solid particles and super absorbent polymers.

Each option provides different challenges to production, conveyance and leak sealing. The options selected have been taken forward for further study and the most promising taken forward to concept experiments.

#### **Liquid Particles**

This work has formed the majority of development work and experimentation. Liquid aerosols are the simplest to produce, as there is a wide range of equipment designed for aerosolising liquids off the shelf. These address many types of liquids and produce aerosols of different particle sizes.

Initially the general behaviour of aerosols have been studied in concept tests using a fog machine to produce the aerosol. Further work has examined the behaviour of viscous aerosols in particular glass aerosols.

#### **Composite Particles**

Two options were taken forward to concept tests:

- Solid bubbles
- Foams

The solid bubbles were investigated as a delivery mechanism concept and the foams as a general initial investigation. Neither of these options are true aerosols but both provided interest and could yield results of value.

#### **Solid Particles**

A number of different solid powders have been sourced and examined in relation to aerosolisation. At the initial stage the particles were not chosen to be tacky more to study the possibility of aerosolisation and conveyance of powdered solids with a view to utilising them in sealing applications.

#### **Super Absorbent Polymers**

This option and other similar systems were selected to be carried out alongside general aerosol research to support additional project work carried out in the 'Gas Polymerisation' project which has identified an opportunity to remotely polymerise vapours using moisture initiators.

#### 3.4 Sealant Specification

At this stage the options are too wide ranging to form a detailed sealant specification, however certain general requirements can be identified for the test requirements of any sealant for the proof of concept and general testing, these are:

- Be able to be aerosolised
- Be able to flow thorough a representative pipe network without significant deposition on the pipe walls, or with control of deposition
- Be able to be driven to and out of a leak
- Be able to be detected in the pipe and at the leak site
- Be able to able to agglomerate at a leak site and to seal leaks of representative size and withhold representative pressure.

#### 4 WP2: Deployment Methodology

This chapter examines options for the implementation of aerosol sealing it looks at different mechanisms how each element may be achieved and lays out the groundwork for the experimentation section of the project. This work package also contained the large element of subcontracted work carried out at the University of Bristol by the Bristol Aerosol Research Centre (BARC). The BARC work examined the evaporation rate of various aerosol particles laying down the groundwork required for designing aerosols of different chemistries. The Bristol work culminated in collaborative testing at Steer where the theory was put into practice with leak sealing tests.

#### 4.1 Aerosol Deployment

The deployment of an aerosol has been broken down into the following stages:

- Generation of the aerosol
- Delivery into the flow stream
- Transportation to the leak site
- Deposition of the first particles on the pipe surface at the leak site
- Accumulation of further particles on top of already deposited particles
- Agglomeration of the particles to form a cohesive seal

These stages are discussed in turn below.

#### Generation

A wide range of equipment exists for the generation of aerosols many of which are beyond patent protection and/or commercially available. The main types are described below.

#### Flame reactor

These systems are used to generate solid particles from a heated gas stream and are predominantly used to produce nano-scale particles in a narrow size range<sup>15</sup>. It is unlikely Steer or its partners would produce such particles but rather would purchase the material and re-aerosolise by another means (see below).

#### Nozzle

The simplest means of producing aerosols is by driving a liquid, including liquids with solid suspensions in them, through a narrow opening i.e. a spray nozzle. This method can generate droplets reliably down to 100µm, the exact limit being liquid dependent. Variation in size may be greater than for other methods but the equipment is cheap and inexpensive.

The best modern examples of this simple device are push-top perfume bottles. However, the most familiar example is the so-called aerosol can in which the spray is augmented with propellant. The propellant provides the driving pressure but more importantly encourage expansion and rupture of the droplets at the nozzle to enhance dispersal and form smaller particles.

#### **Ultrasonic Nozzles**

Ultrasonic nozzles enhance the basic spray nozzle by exciting it ultrasonically. The sound waves break-up the liquid stream meniscus resulting in the generation of very fine droplets. A higher frequency (on the order of 100 kHz) produces the finest droplets down to as little as 5 microns. The equipment has a reputation for producing very narrow bands of droplet size. Alternative ultrasonic nozzles have a resonator in front of the conventional nozzle which generates a field of high frequency sound waves which further break up droplets, resulting in finer particles.

<sup>&</sup>lt;sup>15</sup> http://www.umich.edu/~elements/5e/web\_mod/aerosol/index.htm

#### Atomiser

The next simplest means of producing aerosols is by drawing a liquid, including liquids with solid suspensions in them, out of a fine capillary by the action of a fast flowing gas stream across its end. This method can generate droplets reliably down to  $10\mu$ m, the exact limit again being liquid dependent. Variation in size may be greater than for other methods described below but the equipment is cheap, inexpensive and can be charged without specialist equipment.



Figure 13: An atomised liquid aerosol generated from an artist's handheld airbrush

The best modern example of this simple device is an artist's air brush. 100 years ago, when spray devices were cruder, perfume bottles used atomisers with the gas stream provided by the air within a squeezable rubber balloon. Modern nozzles suffice in perfume bottles today.

An atomiser, or pump driven nozzle, should prove sufficient for the early concept testing of liquid systems and most solid systems too.

#### Nebuliser

Nebuliser is a term predominantly used in medicine and indeed there are a range of mechanisms employed under this name including jet, mesh and ultrasonic types. Nebulisers are used to deliver drugs to the lungs requiring that they reliably produce an aerosol of consistently sub 5µm droplets. Nebulisers again only work with a liquid, but the liquid may hold a suspension of solid particles.

- Jet nebuliser: similar to the atomizer mechanism but using a higher velocity gas stream.
- Ultrasonic nebuliser: excitation of a piezoelectric oscillator in contact with a liquid reservoir generating ultrasonic waves that allows droplets to break away from the reservoir surface. The original work has matured beyond patent protection.
- **Mesh nebuliser:** Ultrasonic excitation of mesh in contact with the surface of a liquid reservoir forcing tiny droplets through the mesh holes into an aerosol. The original patent for this device was filed in 2005.

#### Electrostatics

Electrostatics is a means to augment aerosol generation techniques rather than a mechanism in its own right. The approach works by charging the droplets as they are created so that they repel one another and resist coalescence. This helps to generate a finer and narrower band of particle sizes. Concurrently the target being sprayed by the droplets is earthed or oppositely charged to allow the droplets to discharge and coalesce, and to prevent the surface becoming charged and repelling the approach of further droplets.

#### Delivery

The delivery of the aerosols into the pipe is envisaged to be feasible by injection of the generated aerosol through a port or tapping on the side of the pipe. The generation itself would be undertaken in a chamber to which gas is fed in so that the generated aerosol is swept through the port.



Figure 14: The basic configuration of aerosol generation and delivery into a pipe

However, it may be later identified in some applications that it is advantageous to be able to deliver the aerosol in a concentrated form to a specific point in the network distant from where access can be gained. In its most conventional form this could be a robotic platform carrying the aerosol generation equipment and delivering the sealant into a narrow side branch which the robot cannot navigate.

#### Transportation

The aerosol must be transported through the pipe without internal manipulation if the ability to deliver an aerosol sealant through a port or tapping is going to be effectively exploited. This will require the aerosol to:

- Distribute throughout the flowstream rapidly after delivery into the pipe
- Carry along straight lengths of pipe, ideally under either lamina or turbulent conditions, with minimal deposition
- Transit around tees with minimal loss into blind branches
- Transit around tees and elbows without disrupting the even spread of aerosol particles across the cross section of the flowstream

#### **Deposition, Accumulation & Agglomeration**

To form a seal individual aerosol particles need to be able to

- 1. stick to the surfaces inside a leak, i.e. to deposit,
- 2. and stick to one another, i.e. to accumulate,
- 3. and then to form a cohesive gas tight seal i.e. to **agglomerate**.

#### Deposition



Figure 15: Deposition of the first (green) aerosol particles upon the pipe wall and upon debris (dark grey) which was already adhered to the pipe wall. The dividing streamline indicates the portion of flow drawn into the leak before it can flow downstream

The strength with which a particular particle sticks to a particular surface inside a leak will not be a set value, but will fall in a range. This range will be widened by variations in the particle (e.g. diameter) and in the leak surfaces (e.g. roughness, contamination). And this range will be further widened by the variability in the gas (and liquid if present) around the particles: even to the extent that the condensation of liquid out of the gas (e.g. humidity) has a major influence.

The dust and debris present in gas pipes, not to mention the debris left over from the formation of the leak path (e.g. corrosion, cracking) will inevitably mean that the leak location where the seal must form will be contaminated on the nano and micro-meter scale. This means that the aerosolised particles must be able to either adhere the contamination to the pipe wall or rely on the contamination already being adhered. Remarkably the later may well be the case, "small particles (of under 10 microns diameter) stick so firmly to surfaces that forces corresponding to accelerations of the order of 1,000-10,000g are incapable of overcoming the adhesive forces"<sup>16</sup>. This force is in fact the combined result of molecular forces, capillary forces, self-induced charge differentials, imposed charge differentials and tackiness. It should be noted though that this force drops dramatically for larger particles.

#### Accumulation



Figure 16: Accumulation of the (green) aerosol particles upon those particles already deposited on the pipe wall and upon debris (dark grey). The dividing streamline has shifted as the aerosol begins to congest the leak passage. Note that deposition continues in areas not previously impacted by aerosol particles.



Figure 17: Accumulation of the (green) aerosol particles continues and the dividing stream line continues to move. Accumulation of aerosol is also occurring away from the leak site.

Like particles attract most intensely and, given the surface contamination in pipes, it is likely that the particles, especially if they have a solidifying liquid component, will stick to one another more strongly than to the leak

<sup>&</sup>lt;sup>16</sup> Adhesion of dust and powder, A.D. Zimon

surfaces. This may lead to the initially deposited material breaking off and, depending on the conditions, some of the surface contamination also to provide a cleaning effect. The strength with which solid aerosol particles stick to one another allows them to build up successive layers that progressively protrude further from the surface and thus bridge substantial gaps. Indeed this is the mechanism by which filters work – not by providing holes too small for the particles to pass through but providing a large surface area to which particles attach and then to which further particles are attracted and more strongly attach<sup>17</sup>. This phenomenon is clearly in action in Modera's patent, where the particles are described as being of up to 100µm and bridging holes of at least 3mm.

Condensation of water, or indeed many liquids, can assist adhesion of particles to one another by effecting the charge, providing capillary action or increasing the effective size of the particles. Thus liquids which are not themselves adhesives may assist in the accumulation of seal even if they later evaporate. Indeed an evaporating liquid has some attraction as a temporary 'glue'. Such a liquid would encourage development of seal before a curing mechanism can take effect whilst ensuring deposits elsewhere in the system will later, in the absence of the curing mechanism lose cohesion and disperse in sizes that will not block burners. The gas pipe environment provides a far more controlled humidity environment than found in HVAC ducts, and water vapour is likely to be a useful and important tool in tuning and optimising aerosol sealant processes.



#### Agglomeration

*Figure 18: Accumulation of the (green) aerosol particles continues at an ever decreasing rate in proportion to the distance of the dividing streamline from the pipe wall upstream of the leak.* 



## Figure 19: Agglomeration of the (green) aerosol particles (triggered by time, pressure loading or some property of the pipe wall material) causing an abrupt cessation of the final leakage and formation of a cohesive seal.

Rigid particles will not form a gas tight seal, as they will leave packing voids between them once they touch. However even flexible particles or liquids will not form a gas tight seal of a leak greater than a single particle.

<sup>&</sup>lt;sup>17</sup> Page 304, Adhesion of dust and powder, A.D. Zimon

This behaviour, a result of falling flowrates through the leak bringing falling amounts of sealing particles towards it, requires that the residual leak rate be negligible or that the aerosol material undergoes a process triggered in the leak location which allows the particles or droplets to agglomerate. The phenomenon that might allow this are presented in the table below.

Phenomenon	Particle type applicable to	Example
Collapsing	Solid	Collapse of a congested filter
Swelling	Solid (+ Liquid)	Nappies
Merging	Liquid	Water droplets on glass
Reacting	Solid or Liquid	Two part foam systems
Clogging	Solid + Liquid	Immobilisation of colloid under shear

#### Figure 20: Agglomeration Phenomenon

#### 4.2 University of Bristol Work Proposal

Discussions were held with Prof. Jonathan Reid at Bristol Aerosol Research Centre (BARC) which resulted in a collaboration between BARC and Steer for this project.

#### **Work Context**

The delivery of sealant to a leak site using an aerosol source would have the potential benefits of:

- autonomous identification of the leak site through conformity of particles to the gas streamlines during transport until in the near vicinity of a leak where the inertia of large particles may be exploited;
- the delivery of particles of a directly to the leak site to provide pre-defined structural integrity;
- the delivery of multiple reagents in sequence including the possible introduction of aspherical high aspect ratio particles to provide an initial scaffold for the seal;
- the potential to deliver and co-locate reagents at the leak site prior to triggering a response.

Shown schematically in Figure 21, a sequence of processes can be delineated in the process of delivering and activating aerosols to seal a leak, specifically the:

- 1. Generation of aerosol of the correct size, any required pre-conditioning and their introduction into pipe.
- 2. Changes in size and composition during transport to the leak site and the possibility of exploiting these in the tailoring of the properties of the aerosol (e.g. formation particles).
- 3. Impaction and efficiency of forming a primary seal.
- 4. Possible enhancement of impaction through selecting particles of appropriate morphology.
- 5. Triggering a response to optimise the seal.



Figure 21: Steps in the delivery and activation of aerosols as a sealant

BARC proposed a series of studies to explore methods for tailoring the production and delivery of aerosol of the optimal size, composition and phase (a solid, liquid or amorphous phase) to the leak site. The focus was on generating liquid aerosol, principally because their properties can be more readily tailored than solid aerosol, particle charging can be avoided, and more constant dispersions can be achieved.

#### **Objectives**

The objective was to demonstrate that aerosol of **sector** (e.g. solid instead of liquid), composition (e.g. wet instead of dry) and size can be delivered to a leak site through a combination of single-particle measurements and simulations of aerosol processing, starting in all cases with liquid solutions. In addition, support was to be provided for preliminary measurements using a test-rig simulator and for assessing activation strategies for triggering aerosol response.

#### **Underpinning Science**

In recent years, BARC have developed a range of unique experimental tools to measure the evolving size, composition, viscosity and phase of aerosol particles over timescales spanning from  $10^{-3}$  to  $>10^5$  s. Of principal relevance to this proposal, individual liquid droplets can be captured in a gas flow within 0.1 s of generation and their time-dependent size measured during evaporation. Droplets are in the radius range 3 to 50 µm and their size can be determined with high accuracy (±100 nm in the case of an electrodynamically trapped droplet and ±1 nm for an optically trapped droplet). These tools have allowed the exploration of the underpinning areas of aerosol science that have been used to tailor the properties of aerosol for sealing leaks.

- BARC have shown that the evaporation of water from aerosols inherently solid particles. They have shown that the size of particle and the time at which it forms a state can be tailored depending on the initial size of the droplet when generated and that the particle at any time can be varied depending on the chemical components used.
- Regulation of water loss by trace additives: It has been shown that trace additives can facilitate control
  over the rate of water loss from a droplet. The addition of <1 mM concentrations of long chain
  alcohols can delay the mass flux of water from an evaporating droplet by >5 orders of magnitude. In
  addition, it has been shown that the amount of water retained within the particle can be controlled by
  the addition of polymeric additives to aqueous aerosol, with amplified effects beyond those expected
  from thermodynamic models.

The understanding of these aerosol processes was extended through a combination of experiments and modelling tailored specifically to simulate the evolving properties of aerosol during transport in a natural gas flow. More specifically, a study has been carried out of particles of the correct size and composition in a gas phase of the appropriate thermophysical properties for this application. Ultimately, a framework has been provided for tuning the production and formation of aerosol particles of the correct composition, size and phase/viscosity to possess the correct properties on arrival at the leak site.

#### **Environmental Considerations**

The aerosol research group at the University of Bristol have previously demonstrated the use of a unique electrodynamic balance (EDB) technique to carry out highly accurate comparative measurements of the evaporation rates of two coarse mode droplets of different composition. This method can be used to quantify the influence of organic solutes on the kinetics of evaporation / condensation<sup>1819</sup>. The evaporation of one of the aerosol droplets can be used to provide an accurate calibration for the second aerosol type; for measurements close to saturation, the calibration aerosol is a pure water droplet. A dilute solution droplet is injected into a gas flow of chosen relative humidity (RH) from a droplet-on-demand generator. The droplet loses water by evaporation, typically equilibrating to the sub-saturated RH of the gas flow in a few seconds, sufficiently slow to sustain homogeneous composition. During this process the evolving size can be measured with an accuracy of better than  $\pm 0.5$  % and with a time-resolution of 10 ms.

It is understood that the composition of the natural gas flow consists of 90 % methane, 4 % ethane, 4 % nitrogen, 1 % propane and 1 % carbon dioxide. Ideally the droplets would be exposed to the same environment; however a safer non-flammable gas for the initial experiments with similar properties has been used. The heat and mass transport processes that occur during droplet evaporation and the transport of the aerosol to the leak site depend on the thermophysical properties of the gas phase; thus, a gas with similar parameters to the natural gas flow is needed in order to correctly simulate, interpret and predict the aerosol behaviour. The binary diffusion coefficient and thermal conductivity of gases are shown in Figure 22 using methane as a bench mark, the primary compound of the natural gas. All previous EDB measurements have studied evaporation into either humidified nitrogen gas or an air mixture. This work studies evaporation into a dry atmosphere, a much faster evaporation process leading to much shorter droplet lifetimes. Evaporation into dry nitrogen will provide a lower limit on the upper evaporation rate (upper limit on droplet lifetime) with a thermal conductivity less than that that of methane; evaporation into neon gas will provide an upper limit on the evaporation rate (lower limit on droplet lifetime) with the thermal conductivity and diffusion constant both higher than the value in methane.

The baseline pressure is 25 mbar above atmospheric pressure in a gas riser at a temperature between 0 and 30 °C. This marginal increase in pressure will only have a minor impact on the aerosol dynamics and the evaporation timescales. Thus, measurements will be made at 1 bar over the range of gas velocities accessible to the instrument from 0.01 to 0.5 m/s. In terms of temperature, BARC will focus their work between 10 and 30 °C in this preliminary work; when operating the experiment at temperatures below 10 °C, condensation develops on the outside windows of the chamber, reducing the integrity of the elastic scattering pattern image recorded by the camera. Instead of the additional complexity of using purge gas flows for the window mounts, measurements have been restricted to temperatures above 10 °C.

<sup>&</sup>lt;sup>18</sup> Davies, J.F., A.E. Haddrell, and J.P. Reid, Time-Resolved Measurements of the Evaporation of Volatile Components from Single Aerosol Droplets. Aerosol Science and Technology, 2011. 46(6): p. 666-677.

<sup>&</sup>lt;sup>19</sup> Davies, J.F., et al., *Bulk, Surface, and Gas-Phase Limited Water Transport in Aerosol.* The Journal of Physical Chemistry A, 2012. **116**(45): p. 10987-10998.

Gas	Binary Diffusion Constant	Thermal Conductivity
	(cm² s <sup>-1</sup> )	(mW m <sup>-1</sup> K <sup>-1</sup> )
Methane	0.292 (307К)	34.1 (300K)
Carbon Dioxide	0.202 (307K)	25.1 (300K)
Nitrogen	0.293 (298K)	26 (300K)
Oxygen	0.282 (308K)	26.3 (300K)
Helium	0.908 (298K)	156.7 (300K)
Neon	0.378 (300K)	49.8 (300K)
Hydrogen	0.915 (307К)	186.9 (300K)
Ethylene	0.204 (307К)	20.5 (300K)
Dichlorodifluoromethane	0.105 (298K)	9.9 (300K)

#### Figure 22: Table of Thermal conductivities and diffusion constants of some common gasses

#### 4.3 Bristol Work Packages

The programme of work developed in conjunction with the University of Bristol aimed to demonstrate control over the **second**, composition and size of aerosols in a gas phase and over a timescale relevant for the application, allow particles of the optimal rheology and size to be delivered to the leak site.

- Bristol WP1: Modified Water (Trace Additives): Initial, single particle work on modifying water to control the evaporation of the aerosol particles in relation to time. This work also served to set up experimental equipment and understanding of pertinent mechanisms to very dry atmospheres.
- Bristol WP2: Modified Water (Hydrogels): Adaptation of water by combination with Super Absorbent Polymers in an attempt to deliver water to the leak site to provide options for moisture polymerisation at the leak sites.
- Bristol WP3: Fundamental research to take the understanding from WP1 and WP2 and apply it . The control of evaporation enables particles of a specific viscosity to be delivered to the leak site and seal from aerosol particles.
- Bristol WP4: Simulated Delivery Methods and Ensemble Flow Measurements: This is supporting work for WP1-3, use and development of bench top test rigs to be used in the lab to form a body of experiments to support the theory.
- Bristol WP5: Support For Steer Energy Testing and Cheltenham Facility: This final element of the programme allows time to take the work of the lab and implement it in a 'dirty' environment much closer to the systems found in the field.

#### 4.4 Bristol Conclusions

Overall the work with BARC has been very successful, a detailed understanding of the evolution of aerosol particles in a gas stream has been demonstrated and this has been used to inform the experimental work resulting in a method of sealing leaks.

#### Single particle results

The single particle measurements (WP1-3) have shown that it is possible to transport water to the leak site, however further work will be required if it is desired to pursue this line of research.
From WP1 we concluded that the use of organic films to coat water droplets will provide insufficient delay in the evaporation kinetics of droplets smaller than 5  $\mu$ m in diameter. This is particularly true when evaporation takes place in a gas phase of higher thermal conductivity (neon in this case) than previous measurements with nitrogen gas.

Droplets forming hydrogels, WP3, could provide a plausible route to delivering water in liquid form to the leak site. However, refined measurements are required for the systems studied to quantify the volumes of water that could be delivered and the release kinetics of water from the hydrogel droplets once formed.

The delivery of aerosol to the leak sites was identified as highly possible. From single particle measurements of the evolving size of evaporating droplets, it was inferred that particle ) on a timescale of <2s. Above this

timescales shorter than 10 s and 1000 s, respectively. At 10<sup>5</sup> Pa s, particles can be considered to have become form. This provides a possible mechanistic view of what could occur on sealing leaks. Particles will dry to passage down a pipe. On impaction around a leak, they will immediately

a longer timescale. Continued flowing of dry gas will continue to harden the film,

on a timescale of hours.

Thus, based on the conclusions of the single particle measurements (WPs 1-3), it was decided that sealing priority for study in WPs 4 and 5. A sealing aerosol provide the most compelling evidence that leak sealing could be possible, and could provide a one-step solution to leak sealing not afforded by other methods.

#### Aerosol generation and characterisation

In WP4 BARC concentrated on characterising the generation of the second provide the approaches for generation: an airbrush, a nebuliser and an atomiser. Particle mass concentration distributions from the former two were broad and with maximum mass concentrations at 5  $\mu$ m diameter and above (with particle sizes even extending above 10  $\mu$ m in diameter). Not only would this lead to large sedimentation losses in pipes (noting that the particle mass scales with the cube of the particle radius), but it would lead to the generation of large amounts of water in the liquid form which would need to be dried/removed from the second particle in the gas phase relative humidity, compromising the increase in particle in the second partic

during passage along a pipe and the coherence/solidity of any film formed. Particle mass concentrations from the atomiser peaked instead at a particle diameter of ~2  $\mu$ m, reducing the mass of water that must be accommodate by the gas phase by greater than a factor of 15. In addition, particle mass concentrations were stable for long operational times (hours) compared to minutes for other generators (in particular the nebuliser). Preliminary measurements allowed us to characterise the typical time frames for water loss from the particle sizes generated by the atomiser.

#### Leak sealing simulation

In the final work package, WP5, BARC provided equipment and support to the experimental work being carried out in Steer's workshop. This enabled refinement of experiments culminating in the leak sealing tests. This joint collaborative work was very powerful in terms of informing the lab work with real world data and guiding the test programme. Leaks of 0.3 and 3 mm were investigated and leak sealing was achieved for both sizes on timescales of 10 to 40 minutes depending on gas flow

The final testing and leak sealing provided a high level of confidence in the understanding of the aerosol generation and evaporation during the early portion of the flow and the subsequent deposition at the leak site and final leak sealing.

The belief is that the concept of using this method has been proven and that **account of** aerosols can be designed to be deployed in the gas network to remotely seal active leakage in the gas network.

17 March 2016

The full report of the work carried out by the University of Bristol has been made available to SGN in a separate document.

# 5 WP3: Design of Aerosol Numerical Model

# 5.1 Introduction

This chapter outlines the design and initial development of the CFD model used in this work to provide understanding of the aerosol and gas flow and in particular the interaction with the network and leaks. Testing in this manner allows an indication of the way the vast physical variables interact with each other and although in this stage the work was not expected to yield detailed insights into the problem, it is expected to become more valuable as the project progresses to subsequent stages.

The full report carried out by Norton Straw as directed by Steer Energy is available on request.

# 5.2 Set Up

This study has been carried out based on a simplified geometry as shown in *Figure 23*.



Figure 23: Diagram of geometry

Key Points:

- The leak for this geometry is a cylindrical extrusion from the main pipe bore, of constant circular crosssection.
- A model was chosen with 32 mm internal diameter, and focused on a leak of 1 mm diameter and 32 mm length to simulate a thread leak.
- Work has also been included to include cracks of the same equivalent hole area which are sited perpendicular and parallel to the pipe flow as well (0.04 mm by 19.65 mm).
- Pressure was set as 1000 mbar at the leak outlet, and 1030 mbar at the pipe exit, with the inlet pressure set by the flow rate.
- Methane was used as the gas, and water used to model the aerosol particles.
- Particle diameter was set to 10 μm.
- Gravity has not been considered in this study to simplify the model and recognising that the settling velocity for the particles is significantly low.

Four conditions have been assessed representing different flow rates that correspond, in the main bore of the pipeline, to laminar flow (two Cases at different speeds), transitional flow and turbulent flow. The flow rates and speeds (through a 32 mm diameter bore) are shown in

Figure 24 below.

Case	Reynolds number	Average gas speed at inlet [m/s]	Flow rate [m³/hr]	Particulate terminal velocity [m/s]	Ratio of gas speed to terminal velocity
Case 1 – Very low Reynolds number	100	0.05	0.14	0.0054	8.7:1
Case 2 – Low Reynolds number	1500	0.71	2.06	0.0054	130:1
Case 3 – Transition flow	3250	1.54	4.46	0.0054	283:1
Case 4 – Turbulent flow	10000	4.73	13.71	0.0054	869:1

#### Figure 24: Flow Rates and particulate terminal velocities for Analysis

These results show that gravity may have an influence at lower flow rate Cases and should be considered in later stages of the project. For these Cases, in a horizontal pipe and assuming a uniform mean flow speed, particulates would fall a distance of 16 mm (half of the 32 mm diameter pipe) in 0.14 m and 2.1 m respectively. However, to simply evaluate approaches and techniques at this stage of the study, gravity is not considered.

# 5.3 Particulate Dispersion Modelling

A range of multiphase approaches are available using CFD to model the particulates within the carrier phase of natural-gas. To evaluate these approaches a straight section of pipe, 32 mm diameter was modelled, not including a leak, and using a point source for the release of particulates. This model intended to show the expected dispersion of particulates across the width of the pipe from a point location, and to evaluate how the different models account for the particulate phase adhering to the walls.

The approach assessment was made using the turbulent flow case (Case 4), which was selected due to the high level of certainty in there being effective dispersion of particulates across the pipe width. The approaches assessed are:

- Dispersed multiphase model (DMP);
- Lagrangian multiphase (LMP) model;
- Eulerian multiphase (EMP) model;
- Passive scalar approach.

The analysis and results presented above in *Figure 25* show that the Lagrangian and passive scalar approaches have the potential to provide a computational model of the particulate transport in an efficient manner in terms of computational expense and time. These two approaches have been taken forward to be evaluated in a local model around the leak.





Proportion of particulates remaining in flow with distance

Figure 25: Proportion of particulates remaining in pipe, all approaches

It is envisaged that in any future stage of the development project, experimental data will be used to reevaluate these approaches.

# 5.4 Concept evaluation of Aerosol Sealant Modelling

The two chosen modelling techniques were taken forward and looking at the 4 different cases, a number of models were run to compare the number of particles hitting the main wall with those hitting the wall in or around the leak. The development of the results are shown here for Case 1 of the Lagrangian approach.

Initially 10 particles were modelled, released from the centre of the pipe to give an initial indication of the model behaviour.

To determine the positions of where particulates collide with walls, in comparison with other cases, a second scenario was analysed where particulates were been introduced to the model uniformly distributed across the inlet face. Selected particle traces are shown in Figure 26 which shows the majority of particles exiting the leak an summarises the behaviour of the particles in the system.

In the local model turbulent flow develops around the leak causing particulates to collide with the pipe walls. This is shown via a contour plot of the mass flow of particulates at the wall in Figure 27, which shows how, immediately upstream of the leak, particulates are drawn towards the main bore walls and collisions with the wall occur. The highest rate of particulates collide with the walls is a short distance into the leak.



Figure 26: Selected particle tracks coloured by velocity magnitude



# Figure 27: Contour plot of mass flux of particles on pipe main bore and leak walls

The results of the models were then noted for each case and comparisons drawn up for the various cases. These are presented in *Figure 28* for the Lagrangian approach and *Figure 29* for the passive scalar approach.



*Figure 28: Proportion of particulates adhering to or leaving each surface of model, Lagrangian approach* 



Figure 29: Proportion of particulates adhering to or leaving each surface of model, passive scalar approach

A comparison of these modelling techniques shows significant differences in the results of wall collisions at the leak and this would have to be understood in relation to further experimental in order to understand the 'correct' modelling approach (recognising that this might be different for different aerosols and flow speeds, for example).

Using the same cross-section leak area (for the previously used circular leak), models have also been run for rectangular cross-sections (0.04 mm by 19.65 mm) in both parallel and perpendicular to the main pipe flow direction, and carried out solely for the Case 1 flow conditions.

# 5.5 Conclusions

The initial numerical modelling results indicate that the leak-sealing concept is viable and can be captured by a CFD approach.

Two of the CFD techniques assessed are identified as having the potential to simulate, and therefore further develop the Aerosol Sealant technology; these are the Lagrangian multiphase and passive scalar techniques. The two methods predict different proportions of particulates accumulating on the leak path surfaces.

It has been concluded that:

- The trajectories of particulates, for small particulate sizes, will be highly dominated by the flow.
- The flow split between the main bore and leak will greatly influence the likelihood of particulates colliding with the walls of a leak.
- At low pipeline flow rates a greater proportion of flow will travel through the leak and therefore have greater probability of colliding with the walls around the leak which is advantageous to eventually block the leak.
- At very low Reynolds number conditions, the leak geometry has a significant impact. The higher-pressure drop across a narrow and long leak, compared to a circular hole, causes less flow to travel through the leak so fewer particulates to collide with the leak walls.

It was noted that a large proportion of particulates may be lost through deposition on the main bore walls upstream of the leak, or will continue in the flow through the main pipeline rather than being diverted towards the leak.

To gain a greater understanding of the performance of this concept for pipeline sealing further work, initially through comparison with relevant experimental data, will require to be carried out. This would enable further investigation and calibration of the techniques presented in this study and enable better understanding their ability to predict the leak-sealing phenomena to aid the development of this technology.

# 6 WP4: Experimental Set up and Testing

This chapter details the experimental work carried out to test the various concepts under investigation. Many of the sealant options developed in WP2 were explored further prior to formal investigation to gain an understanding. A few select cases representing the more promising options were then promoted to concept testing and the most successful of those was taken to detailed testing eventually reaching leak seal tests.

# 6.1 Concept Tests

The concepts used for initial study are as follows:

- **Solid bubbles:** this was an early test for a novel delivery system suitable for many different aerosol types. This was not taken forward.
- Liquid aerosols: these are the most common aerosol type and the easiest to produce. Most work was carried out on this option and it has progressed the furthest. Experimental work has also been carried out in conjunction with University of Bristol. Many options are available for aerosolisation and such aerosols are simple to flow through a pipe network. Leak sealing has also been achieved with very low volumes of sealant required to remotely seal a leak.
- **Solid aerosols:** this work provided useful information for augmenting the liquid aerosol work. This was not taken to detailed testing but has been used as an information gathering exercise.

All of the investigations into the concept tests followed the same basic procedure:

- 1. Can the concept aerosol be produced?
- 2. Can the concept aerosol be conveyed through a pipeline environment?
- 3. Can the concept aerosol be used to seal a leak?

During the experimentation, the most promising candidates have been progressed to later stages whilst lesser candidates are left and the information noted for future use.

#### **Solid Bubbles**

The aim of this test was to produce a delivery system suitable for many different types of aerosol (demonstrating its use as an 'enabling technology' rather than an aerosol itself).

#### Solid Bubble tests

For example, an acetone solvented plastic (polyvinyl acetate) that can be blown into bubbles is commercially available as "Magic Plastic". Bubbles of this were blown with helium to assess if a free floating bubble could be produced. The method of inflation generally leaves an un-inflated portion or sprue of the material. This sprue was nearly always comparatively massive compared to the bubbles. One of these bubbles (8mm diameter) was then burst, the sprue cut off and the bubble surface rolled into a ball. This diameter of the ball was measured and then the mass and buoyancy of the bubble determined. It was found that the bubble would have been neutrally buoyant without this sprue feature.



*Figure 30: A polyvinyl acetate bubble with sprue. Inset are images on the same scale of the bubble and the sprue separated and rolled into spheres.* 

A preliminary assessment has been undertaken that has shown bubbles could have the necessary physical properties to carry significant quantities of sealant through a riser to a leak site.

The calculation was repeated for a range of bubble sizes that the manual blowing arrangement could not provide, to provide an indication of how much sealant a neutrally buoyant bubble could carry as its surface film within a gas stream (see Figure 31).

Methane	0.66	kg/m3
Helium	0.164	kg/m3
Buoyancy	-0.496	kg/m3

Plastic 1190 kg/m3

Bubble internal diameter	mm	0.5	1.0	2.0	4.0	6.0	8.0
Bubble gas volume	m <sup>3</sup>	6.5E-11	5.2E-10	4.2E-09	3.4E-08	1.1E-07	2.7E-07
Bubble gas buoyancy	kg	-3.2E-11	-2.6E-10	-2.1E-09	-1.7E-08	-5.6E-08	-1.3E-07
Allowable bubble surface mass	kg	3.2E-11	2.6E-10	2.1E-09	1.7E-08	5.6E-08	1.3E-07
Bubble surface area	m²	7.9E-07	3.1E-06	1.3E-05	5.0E-05	1.1E-04	2.0E-04
Allowable bubble surface thickness		0.02	0.07	0.14	0.28	0.42	0.50
Allowable bubble surface thickness	μm	0.03	0.07	0.14	0.28	0.42	0.56
Equivalent diameter of burst bubble film	mm	0.04	0.07	0.15	0.30	0.45	0.60
Ratio film thickness : bubble diameter	-	0.01%	0.01%	0.01%	0.01%	0.01%	0.01%

# Figure 31: Bubble Volume Calculations

The ability to vary the wall thickness of the bubble gives the potential to change from a situation of targeting the bursting and deposition of sealant at the leak site, to using a large 'instable' bubble to provide wall wetting. For example, a 1mm diameter solid liquid particle would be constructed of around 0.3 mm<sup>3</sup> of liquid;

assuming a wall thickness of 500 nm for the bubble, the bubble would have to be around 600 mm in diameter to carry the same volume. It therefore has potential for wall wetting far superior to that of a single particle.

It is clear that the sizing of the bubbles is vital in order to adequately transport and entrain the bubbles into the leak if this is the planned use. Both the standard motive forces and gravity will play a significant role in determining the optimum size, along with volume of fluid required to be deposited at the leak site.

#### Solid bubble conclusions

This work has shown to be sufficiently challenging for the first task of generation that it was not pursued further.

#### **Liquid Aerosols**

This group of aerosols was expected to be the easiest to generate and convey aerosols. Initial tests have been carried out to observe the conveyance of the aerosol and the detection systems which may be used. These tests have been as much about developing the test equipment and understanding of the tests as they have about developing the technology.

#### **Generation Equipment**

A commercial fog machine is being used to generate highly visible, persistent aerosols. This is particularly effective for visualising the flow of aerosol through the pipe system and out of leakage sites.



Figure 32: Fog Machine feeding into the loop

The fog machine functions by pumping a small amount of a 'fogging liquid' into a heat exchanger set at 200-300°C. The fluid atomises inside the heat exchanger and exits as a jet of vapour. On exiting the machine the mixture mixes with cooler air and the vapour condenses, resulting in a thick visible fog. The size of the particles will depend somewhat on the operation of the machine, and different machines produce different sizes of particle. Cheaper fog machines such as the one being used generally produce fog with particle sizes with a mass median diameter of between 1 and 10  $\mu$ m. Fogging liquid generally comprises distilled water with an addition of components such as glycerol or glycol to slow down the evaporation of the water and hence prolong the vapour cloud.

The fogging machine used provides a good supply of a visible aerosol which can be pumped throughout a pipe network. It does not have the ability to significantly vary the liquid nor does it have the facility to change the particle size.

The aim of the first concept tests was to demonstrate that an aerosol could easily be produced and that this aerosol could be conveyed through a representative pipe system and detected at the end of the pipe system.

#### **Flow Equipment**

For this first simple test a loop of pipe was constructed as shown in Figure 33. The fog machine was used for the aerosol generation and a fan was installed into the loop to provide motive force for the flow. The vane anemometer was used to measure flow speed for different fan voltages. The loop was constructed with tees and bends and clear pipe sections to enable the aerosol to be visualised in the pipe system.



Figure 33: The first loop utilised elbows and tees

## **Initial Tests**

From the outset the tests showed the aerosol visibly passing through the clear sections of the loop and out of the end of the loop. The aerosol exiting the loop gives an indication of the degree of turbulence of the flow as can be seen in Figure 34. The left hand side of the figure shows the aerosol exiting the pipe at a speed of 0.5 m/s. The Reynolds number at this speed is 1416, which describes laminar flow as indicated in the photo. The right hand side of the figure shows the aerosol exiting at 3.5 m/s, the Reynolds number is now 9913 indicating Turbulent flow.



Figure 34: Aerosol exiting pipe at 0.5 m/s and 3.5 m/s

The loop was then extended by the addition of a 30 m section of pipe, to demonstrate that the aerosol could easily travel through longer sections. This was achieved without any visible reduction in the aerosol.

It was noted that running with the aerosol for long periods of time resulted in the aerosol coating the walls of the pipe with a fine condensation. The aerosol produced by the fog machine is designed to be persistent so it should be expected to remain in the atmosphere for up to 10 minutes. During this time at a modest 1 m/s flow the aerosol would penetrate 600 metres of pipework. A penetration of 1 km is therefore easily achievable for an aerosol of this nature.

The outflow of the aerosol from leaks could be controlled by internal pipe pressure. Aerosol jets exiting test leaks were clearly visible, there was no indication of aerosol building up at the leak site however during the initial tests.

During these tests it was noted however that the aerosol vapour had a tendency to travel in the lower half of the pipe, especially in the head and the tail of the slug of vapour, Figure 35. As the velocity was increased then this effect became less noticeable. This is something to be aware of going forward and it is something to note with the use of fogging technologies, they will have a tendency to dominate the lower half of the pipeline in preference to the top half which could make sealing on the upper portion of the pipe challenging. This is likely to be the case for any aerosol where the particles are significantly heavier than the gas suspension medium.



*Figure 35: Detail of Vapour Travelling in Lower Portion of Pipe (RHS) and comparison with empty pipe (LHS)* 

## Liquid Aerosol Conclusions

This is a fundamental technology which should be taken forward, it was expected that this technology would pass the generation and conveyance / detection tests. The challenge will be on leak sealing which is carried out in the detailed testing.

## **Solid Aerosols**

Solids can bring a number of advantages to the table: they also bring significant challenges. The creation of a solid aerosol, if carried out without the aid of a liquid base requires either small particles to be created and aerosolised in one operation or the aerosolisation of existing small solid particles. If the particles are designed to agglomerate then one would expect a container of unaerosolised particles to instantly agglomerate.

The experimentation of solid aerosols has therefore concentrated in the first instance on solid particles in powder form which are not designed to agglomerate. The aim is to investigate the feasibility of firstly aerosolisation and secondly conveyance before looking at leak sealing aspects. A range of particles of different types have been sourced and investigated for aerosolisation.

## Examination of the different particles

The range of powdered materials were briefly analysed under the microscope. The first point of note is that all of the particles are large compared to the liquid aerosol particles investigated to date which have been in the region of 0.5 to 10 microns. The visual analysis is presented in Figure 36, Figure 37 and Figure 38. The overlay scale is in 50  $\mu$ m divisions. The 0 to 20 on the scale therefore shows a 1 mm total gap. The different substances have different ranges of particle sizes and so will be relevant in different sealing mechanisms.



Figure 36: Glass Bubbles, 'Fillite' Hollow MicroSpheres, Milled Glass Fibres



Figure 37: Powdered Rosin, Fumed Silica, Talc



Figure 38: Powdered Sugar, Flour, Cornflour

The visual inspection of the various solid particle systems has enabled an estimation of the range of particle sizes to be carried out, this is given in Figure 39. The majority of these solids are large in comparison to the liquid systems tested, however it has been possible to suspend these particles in gas flows.

Material	Smallest Observed Particles	Largest Observed Particles
Glass Bubbles	20 µm	100 μm
'Fillite' Hollow Spheres	40 µm	200 μm
Milled Glass Fibres	8 x 10 μm	8 x 500 μm
Powdered Rosin	20 µm	100 μm
Fumed Silica	20 µm	150 μm
Talc	5 μm	40 µm
Powdered Sugar	5 μm	50 μm
Flour	10 µm	30 µm
Cornflour	5 μm	10 µm

# Figure 39: Estimation of range of particle sizes for the powders

#### Aerosol generation from powders

A test system was created, illustrated in Figure 40 to aerosolise the powders under test. This was achieved using a vertical section of pipe with a gas jet placed in the bottom of the pipe. The gas jet had two purposes, firstly to break up the solid mass of particles into individual particles, in particular breaking up clumps of particles. The second purpose was to provide a flow of gas to a subsequent pipe providing onward flow of aerosolised particles.



Figure 40: Powder Aerosol Generation

#### Conveyance of Solid Aerosols

Simple conveyance of solid aerosols was possible; however there was a tendency for the solids to drop out of suspension over long runs when the flow rate was low and in the laminar flow regime. The particles had a tendency to stick to the pipe walls, more on the lower half due to the drop out, and this tendency was much more marked when moisture was present in the system.

#### **Conclusions of Solid Aerosol Concept Tests**

The use of solid powders to generate an aerosol has been demonstrated and it has been possible to convey this aerosol through a pipe network. It should be noted that this has been shown for non-sticky particles, sticky or tacky particles would provide a much greater challenge to the process of aerosolisation, indeed a separate means of aerosolisation would be required. One such means would be suspension and aerosolisation in a liquid which subsequently dries leaving a sticky solid. The study of solid particles is taken forward as a fall back opportunity in the next stage of detailed testing and may subsequently be used to support and augment a liquid system.

#### **Concept Test Conclusions**

The overall concept test conclusions are as follows:

- Solid bubbles are not being taken forward as we experienced significant challenges in progressing this work.
- Solid aerosols could provide support to other work. It has been possible to aerosolise solid powders and convey them through the test loop, however the particles were not optimised for flow or leak sealing. This may warrant further investigation in the future as an addition to liquid aerosols. Solid tacky aerosols also follows existing work by third parties and as such is less attractive in generation of alternative methods.
- Liquid aerosols have shown the most promise in the conceptual testing. Liquid aerosols are directly which are being tailored compatible with the work being carried out at BARC on for use in leak sealing tests.

It was therefore decided to concentrate on liquid aerosols,

in the next phase of experimentation: detailed testing. The aims of the tests are to bring together the theory developed to date and the practical experience from the concept tests to optimise the behaviour of the liquid aerosol such that it becomes efficient at leak sealing.

# 6.2 Detailed Testing

Following on from the concept testing, detailed testing aimed to take the preferred viscous glassy aerosol system and examine in more detail methods of generation, conveyance and interaction with leaks with an overall view to sealing leaks.

A wider range of equipment was examined for suitability in terms of generating and conveying aerosols through pipe systems to leak sections.

#### **Aerosol Generation Equipment**

#### **Ultrasonic Nebulisers**

Ultrasonic nebulisers use piezo electric transducers to excite a pool of liquid producing a vapour from the ultrasonic shock waves passing through the liquid. This type of nebuliser is able to vaporise low viscosity products such as water, but the addition of heavier or more viscous components significantly reduce the amount of vapour produced. It is however able to aerosolise low concentration sugar solutions. The ultrasonic nebuliser produces a much smaller amount of vapour than the fog machine, however it is able to vapourise solutions of other materials which are heat sensitive (including diluted syrup solutions).





Figure 41: Ultrasonic Nebuliser

#### Ultrasonic Mesh Nebuliser

The ultrasonic mesh nebuliser works by vibrating a mesh with tiny holes in. The ultrasonic vibrations cause small droplets of liquid to be forced through the mesh, resulting in a stream of controlled nebulised particles of liquid. The vibrating mesh nebuliser is better able to nebulise sugar solutions than the basic ultrasonic nebuliser.



Figure 42: Ultrasonic Mesh Nebuliser

#### Artists Airbrush

An airbrush works by passing a stream of fast moving compressed air through a venturi, which creates a local reduction in air pressure that allows paint to be pulled from an interconnected reservoir at normal atmospheric pressure. The high velocity of the air atomises the paint into very tiny droplets as it blows past a jet and needle metering component. The brush is operated by a dual action trigger / lever. Pressing the trigger down increases the air flow, pulling the trigger back increases the paint flow. A third adjustment is possible: the supply pressure of the air. These three settings have the ability to change the size of the different droplets. The airbrush has the advantage that is has been designed to operate with higher viscosity fluids such as paint. An example of the construction of the airbrush is shown in Figure 43.



Figure 43: Commercial air brush

#### Atomiser

The work combined with Bristol has utilised an atomiser to produce the aerosol this works on a similar principle to the airbrush, by having a flow of gas drawing the liquid up into the system and producing an aerosol. The aerosol is generated in a chamber which allows the heavier, larger particles to return to the liquid supply. The atomiser produces more consistent and smaller particles than the airbrush.



Figure 44: Liquid Atomiser

## Flow and Pressure Generation and Measurement

Flow is generated in the test equipment by a combination of fans and the pressure supplied by the generation equipment. An example of a stack of fans is given in Figure 45. Each fan generates a pressure differential producing flow in the system. The stack of fans generates a bigger pressure differential than a single fan.



Figure 45: Stack of 12 V, 40 mm fans

A range of measurement equipment was used to measure the speed of the flow within the pipe systems. Simple measurements were made with the vane anemometer, Figure 46 left hand side. The hot wire anemometer was then used as a calibration device as this is more sensitive. It should be noted that the anemometers have a minimum as well as a maximum range so the sensitivity of the measurements are less at very low flows.



Figure 46: Vane anemometer and hot wire anemometer used for flow measurement

The anemometers and fans were included in the test loops as required by the various tests. Figure 47 shows the equipment configured for use in the test loops along with a digital pressure gauge. The gauge used is a 2.5 bar gauge with 1 mbar resolution. An additional manometer was used with a 50 mbar full scale and 0.01 mbar resolution.



Figure 47: Fans, Anemometer and Pressure Gauge configured for use in a test loop

# **Gas Supplies**

The domestic gas supply is, on the whole, a dry source of gas. Moisture ingress can occur, however this is prevented where possible with the result that normally the gas flow has very low levels of humidity. Recognition of this aspect of the application is very important as the nature of any liquid aerosol will rapidly

change by evaporation. Aerosol particles will dry much more rapidly in a dry atmosphere than a humid one. As the tests progressed the need for a dry gas supply was more and more important.

Initial testing was carried out using air from the atmosphere either unconditioned from the workshop in raw or compressed form.

These included the fabrication of a small environmental

chamber for mixing of aerosols, drying of air and inclusion of additional drying units to ensure the air supply is dried. These units included de-humidifiers and desiccant materials.

For final testing, nitrogen cylinders were used to provide a dry supply of gas closer in humidity nature to the natural gas supply.

# 6.3 Leak sealing tests

Leak sealing tests were carried out with the liquid aerosols only. The majority of the work was carried out

This is easily dissolved in water and produces a safe aerosol which increases in matches the single particle work carried out by the University of Bristol and this builds on a large body of expertise in the research group at Bristol. The majority of the aerosols used for the tests have been generated using the airbrush or the atomiser. The final successful leak sealing tests utilised the atomiser for aerosol generation.

Tests leading to the eventual leak sealing are presented here in four groups of tests which chart the progression of tests and summarise the findings from each test.



Figure 48: Progress of Testing Results

# Group 1 Tests

This group of tests aimed to convey the aerosol to the leak site, detect it passing through the leak and to observe general behaviour.

Key results were the successful generation and conveyance of a **mattern** aerosol. Early indications of the potential for the build-up of particles at the leak site were seen and these particles coalesced to form a larger object.

# Test #1.1

The first loop was setup as shown in Figure 49, it is a simple fan driven line with the airbrush injecting flow into a straight clear section of pipe.



Figure 49: First Test Loop

Test #1.1 details included:

.

- 1.5 mm leak in clear test section on base of the pipe
- 1 m between aerosol and leak
- Air compressor set to **20 psi**
- The anemometer indicated a flow of **1.8 m/s**

The loop was run for a period of approximately 20 minutes and during this time a number of observations were made and recorded. The vicinity of the pipe immediately after the airbrush was coated with course beads of liquid, shown in Figure 50, and this liquid also collected in the base of the pipe, running down through the fans.



# Figure 50: Liquid deposits in first portion of the pipe

Further down the pipe the liquid tended to collect on the bottom of the pipeline, as can be seen on the left hand side of Figure 51. After the test a partial blockage of the leak was witnessed.



# Figure 51: Beads of liquid on bottom of pipe (LHS) and a partial blockage of the 1.5 mm hole (RHS)

The partial blockage of the leak seemed to be of a crystalline nature but this was not confirmed in this first test. The results were seen as encouraging however but it was clear that the system had too much fluid present. The distance from the injection point to the leak was approximately 1 metre so a speed of 1.8 m/s resulted in a transit time of less than a second. It was decided therefore to increase the length of the loop to try to reduce the deposition of fluid onto the pipe wall adjacent to the leak.

#### Test #1.2

Additional pipe sections were added to the loop so that the amount of liquid collecting on the pipe walls would be reduced. The modified loop is shown in Figure 52. This loop was run under the similar conditions to test #1.1:

- 1.5 mm leak in clear test section on base of the pipe
- 4 m between aerosol and leak
- •
- Air compressor set to **20 psi**
- The anemometer indicated a flow of **1.8 m/s**



# Figure 52: Loop used in Test #1.2

Test #1.2 was run for 1 hour and during this time there didn't appear to be a significant build-up of liquid on the pipe. Examination of the pipe at the end of the test did show droplets forming on the lower half of the pipe, these droplets were much smaller than those witnessed in test #1.1. The aerosol could not easily be seen

at the leak site, however when illuminated by a laser the aerosol particles can be clearly seen flowing away from the leak section.



# Figure 53: Aerosol illuminated by Laser

At the end of the test a bead of liquid was seen at the inside of the leak section, Figure 54.



Figure 54: Bead of liquid seen across the leak in Test #1.2

On stripping down the test loop, a large amount of liquid was seen to have built up at the end where the aerosol doubles back on itself. This is like an impingement filter taking out larger aerosol particles. This aerosol flow is turbulent in this pipe at 1.8 m/s with a Reynolds number in excess of 5000 a slower flow may have helped to keep the larger particles in suspension. The longer transit time, of 2 seconds resulted in smaller particles collecting on the base of the pipe; however the particles were still too wet.

Note all aerosols should be tested for travelling round corners as this is prevalent in the riser system. This may be a fine control system for the aerosol prior to entry into the pipeline system.

#### Test #1.3

Test #1.3 used a generation chamber, the aim for this was to spray the aerosol into the chamber and then flow the aerosol out of the chamber into the leak section. This system had a much longer residence time than the simple pipeline loops. The flow measurement suggested a residence time of up to 100 seconds.



Figure 55: Generation Chamber used in Test #1.3

The setup for Test #1.3 was as follows:

- 1.5 mm leak in clear test section on top of pipe
- 0.5 mm leak in clear test section on base of pipe
- Generation Chamber between aerosol and leak section
- **0.6M** sucrose solution (created by dissolving 10 g of sugar in 50 ml water)
- Air compressor set to 20 psi

The drop in sucrose concentration was chosen to match the testing to the work being carried out at the University of Bristol. The loop could be recirculated to allow a body of aerosolised particles to form and be run past the holes multiple times. It was also noted that after the tests were concluded and the aerosol and circulation switched off, the aerosol remained visible in the box (using a laser indicator) for over 1 hour. This indicates sub  $\mu$ m particles are being produced in the system. The bottom of the generation chamber was also wet with the solution so the heavier aerosol particles were dropping out of the system. The test showed droplets of solution depositing of the inside of the pipe walls but there were no signs of blockage of the leaks as shown in Figure 56.



Figure 56: 1.5 mm leak and 0.5 mm leak with droplets on inside of pipe

It was noted from these tests that the lower half of the pipes tended to be coated in a slight deposit. Nearer to the airbrush a significant amount of fluid tended to be collected from larger aerosol particles dropping out of suspension. This corresponded to the majority of the output from the airbrush.

#### Group 1 test conclusions

Group 1 tests have shown it possible to generate aerosols and to convey them around pipeline systems. The result was a very wet deposit on the pipe walls and one which dropped out of suspension at 90° bends.

At this time discussions were held with the University of Bristol on the performance of the system compared to the individual aerosol particle measurements which were carried out in the Bristol Laboratories. This led to changes being made in the loop and a visit from the Bristol team to carry out some detailed measurements in the workshop scenario.

#### Group 2 Test

On 27<sup>th</sup> January 2016, researchers from the University of Bristol assisted in the testing. The need to maintain low RH throughout the tests was highlighted. Two humidity meters were used to give indicative measurements of the RH during these tests.

The challenge of maintaining a dry air supply was met by changing to bottled nitrogen for the gas supply. This matches the nitrogen supply used in the lab at Bristol. The test loop was reconfigured to run at much lower humidity than the previous group of tests.

This loop was a simple single straight pipeline system. A range of leak sections were trialled during the day of testing the most interesting test seemed to achieve partial sealing of one of the leaks.

A key change to the system was the addition of a relative humidity measurement so that the system could be operated at a reduced humidity level. Ideally for these tests the target RH was less than 35%. The aerosol was generated using an atomiser which produces a more precise aerosol size than the airbrush. An additional flow of nitrogen was used to keep the relative humidity below 40%. The atomiser and tee in of nitrogen flow can be seen in Figure 57.

The operating parameters for the loop were:

• 4 off 0.3 mm leaks in clear test section at the top, bottom and sides of pipe

- 4.5 m distance between the aerosol generator and the leak sections
- •
- Flow rate of 0.5 m/s
- Atomiser used for aerosol generation set to 10 psi



Figure 57: Atomiser used to generate aerosol and additional nitrogen feed to control RH.

Sensitive measurement equipment was used to sample and record the aerosol particles exiting the leak section, Figure 58. This enabled the performance of the system to be measured and the effects of changes in the system noted.



Figure 58: Particle counter and Optical Particle Sensor



Figure 59: Optical Particle Sensor taking measurements of aerosol

Data analysis of the work carried out shows a reduction in the amount of particle concentration seen exiting the leak. The analysis, shown in Figure 60, shows an apparent reduction in the size of the leak over the one hour period. Most interestingly the reduction in size is most significant for the 2.2 to 3.3  $\mu$ m size particles whilst the smaller particles exhibited a smaller reduction in number of particles passing through the leak.



Figure 60: Particle Count Analysis of aerosol exiting the leak

#### **Group 3 Tests**

Discussions with the Bristol team highlighted the importance of maintaining the atmosphere humidity to ensure that the aerosol produced is of the correct properties for the second seco

The humidity was now measured using two loggers with external sensors and up to 1 second sample rate. These give good feedback to control the RH of the system during testing.



Figure 61: The modified test loop used for group 3 tests

Key features of the modified test loop were:

- Dry Nitrogen only feed
- Large pipe bore of 102 mm for long transit time
- 4 off 0.3 mm leaks at 3, 6, 9 and 12 o'clock positions
- Sump to catch larger aerosol particles
- Top cap with relative humidity sensor
- End Cap / Vent with relative humidity sensor

The loop was run in a number of configurations, each configuration produced learning for the system operation and the behaviour of the leak sealing solution.

•

The control of the flow through the loop is determined by the pressure used to drive the airbrush, air flow measurements were taken and this used to determine the transit time for the aerosol through the test loop. The operating range produced transit times of between 25 and 60 seconds.

Aerosols were observed lasers or high powered torches, aerosol can be seen exiting the loop and a leak in Figure 62.



Figure 62: Visual indication of aerosol exiting the loop (LHS) and a leak (RHS)

#### Test #3.1

The running parameters for test #3.1 were as follows:

- Injection pressure (20 psi)
- Needle Position (1 turn)
- Loop Differential pressure (**5 mbar**) this was an initial pressure, the operating pressure was varied throughout the testing and the change on the aerosol expelled from the leak was observed.

Key outcomes of the first test was indicative flow of the aerosol throughout the system and blockage of one of the four test leaks.

, which indicates the leak on the base of the pipe has been blocked.

The next stage of testing was a repeat run of this first test with greater observation of the leaks in an attempt to repeat the blocking of one or more of the leaks.



Figure 63: Humidity Log for Test #3.1



Figure 64: Results of Test #3.1

# Test #3.2

This second test was run in a similar fashion to the first test. Operating conditions for the loop were as follows:

- Injection pressure (20 psi)
- Needle Position (1 turn)
- Loop Differential pressure (**1 mbar**), during this test the differential pressure was changed and the effect on the leaks observed.
- •

The loop was run for a one hour period and once again the humidity in the loop with this setup was high, in the region of 90% RH over the test time, shown in Figure 65. The resulting leak blockages are shown in Figure 66.



Figure 65: Humidity measurement of Test #3.2



Figure 66: Liquid blocking all four leaks

The leaks were only blocked when very low pressure was applied to the system. This was achieved by keeping the end cap loose. If the pressure was raised above 1 mbar then the liquid blocking the leaks was forced out of the leaks, opening up the hole.

This effect is shown in Figure 67 which shows the liquid bead on the pipe wall, whilst the leak is flowing.

willist the leak is nowi



# Figure 67: Liquid beads forming outside the leaks

It was clear at this stage that it is possible to deposit aerosol at the leak site, but this deposition either occurred at the exit of the leak, or was forced out of the leak by the pressure. The liquid deposits were not viscous enough to block the leaks and withstand any pressure.

#### Test #3.3

The aim of the next test was to run the system at a highly reduced value of humidity in an attempt to create more viscous glassy particles. The humidity is varied by reducing the jet/needle gap on the airbrush. Running the airbrush at ½ turn open produced a very small amount of liquid so this was the starting point for this test. The operating parameters for test 3 were therefore:

- Injection pressure (20 psi)
- Needle Position (**1/2 turn**) this was an initial value, it was changed during the test to observe the effect on the humidity and the leaks
- Loop Differential pressure (1 mbar).

#### •

The test was started at 18:00 with the needle open ½ turn. It was run in this manner until 18:20. During this time the amount of aerosol produced was very low, it was not possible to identify the flow using either the powerful torch or with the laser.

At 18:20 it was decided to increase the aerosol flow slightly.

. The liquid flow was backed off and the humidity dropped to 40% RH. This course unstable control continued until 18:57 whilst the leaks were closely observed.

Over this test time, no significant deposition on or near to the leaks was observed. The leak section was removed and photographed, Figure 69 (LHS).

. Straight away liquid beads started to form on the exit of the

holes, Figure 69 (RHS).

Header text



Figure 68: Humidity log from Test #3.3



Figure 69: Test #3.3 Low humidity (LHS) High humidity (RHS)

It is clear therefore that the deposition of aerosol occurs when a large amount of aerosol particles are present. With the current system, this is only achieved at high values of humidity. The deposition can be seen by liquid beads appearing on the outside of the pipe adjacent to the leaks.

# Test #3.4

The second humidity logger was now used to assess whether the humidity at leaks was significantly different to the top of the loop.

This test aimed to repeat test #3.3 but once an aerosol was detected, a clear slide was placed below the bottom leak with the hope of catching aerosol particles impinging on the slide. The loop was run under the following conditions:

- Injection pressure (20 psi)
- Needle Position varied
- Loop Differential pressure (5 mbar).
- •

Initially the loop was run with no liquid flow, to purge humidity from the system.

This was run for 5 minutes but after this time it was not possible to see produced aerosol with the laser. The decision was made to increase the liquid amount, the airbrush jet was opened to allow more flow of liquid and the . It was run

like this for 45 minutes. During this time a slide was placed immediately below the bottom leak to catch aerosol particles exiting the leak.



Figure 70: Humidity measurement for Test #3.4

No sealing of the leaks witnessed but a liquid deposit was seen on the pipe wall adjacent to the leak with the slide (#3) in Figure 71, and on the slide itself.



Figure 71: Results of Test #3.4 with liquid deposition on slide (RHS)

# Test #3.5

The aim of this test was to repeat the previous work and to see if any build-up of particles were seen at the leak sites when the system pressure was changed.



Figure 72: Humidity measurement of test #3.5

The system was initially run at low humidity,

Very quickly droplets formed on the exit to the holes. Control of the pressure in the system led to the liquid deposits being forced out of the leaks.

end result was two of the leaks blocked and two beads of liquid adjacent to the holes on the other leaks, Figure 73.



Figure 73: Results of Test #3.5

#### **Group 3 Test Conclusions**

The system had the ability to run at reduced levels of humidity however when running at low levels of humidity the volume of particles seemed insufficient to build up particle deposits at the leak site.

Particle deposition was possible at higher levels of aerosol which resulted in higher levels of humidity.

The
It was clear that the additions were required in the system to allow for better measurement of the pressure in the system and the system and the system and the system and the system are specified with the system and the system are specified with the system and the system are specified with the system and the system are specified with the system are specified withe

Discussions with Bristol suggested that the atomiser would be a better source of aerosol as it produces particles with more consistency than the airbrush.

A further set of tests were agreed with the Bristol team and the test setup was refined for use in these tests.

#### **Group 4 Tests**

Key features of the group 4 tests were further refinements to the pressure measurement using a digital manometer with 0.01 mbar precision. The atomiser was used for the aerosol generation and the aerosol was to be supplemented with an additional flow of dry

For these tests another version of the linear test loop, Figure 74, was used with the aerosol being generated by the atomiser. A secondary flow of dry nitrogen was used



Figure 74: Test loop used for Group 4 tests

## Test #4.1

The loop configuration for Test #4.1 was as follows:

- 4 off 0.3 mm leaks in clear test section at the top, bottom and sides of pipe
- 10 m distance between the aerosol generator and the leak sections
- •
- Atomiser used for aerosol generation set to 10 psi
- Flow speed of 17.6 cm/s generated by the atomiser
- Operating Pressure was set to 10 mbar

The system was set up with the leak section used before however the pressure in the loop was controlled by a larger, 3 mm hole in a section of pipe after the leak section. This acted as a pressure relief for the system such that a significant change in the rate of leakage would result in a corresponding increase in pressure.



# Figure 75: Humidity measurement for Tests #4.1 and #4.2

After running the loop in this manner for 1 hour there was some limited indication of changes at the leak site from the optical particle sensor but no significant evidence of leak sealing. This behaviour continued at a range of ratios of raw nitrogen to atomiser.

The conclusion at this point was that the atomiser did not produce to achieve good leak sealing. The performance was deemed to be due to a complex set of factors including:

the distance between the atomiser and the leak.

gas and



Figure 78: Blocking of the relief hole



Figure 79: Two blocked leaks, two re-opened leaks



Figure 80: The 3 mm leak from the outside and inside of the pipe after absorbing moisture

Further investigation will be required to further

assess this behaviour in the next stage of work.

Test #4.3

The final test was a repeat of the previous test where the relief hole was as much the target leak as the original test section.

# The four leaks on the test section were sealed within a space of minutes **and the section**. The most challenging of the four leaks to seal was the one on the top of the pipe, this leak will have the least amount of aerosol passing through it due to the tendency of the particles to settle towards the bottom of the pipe. This is despite the fact that many of the particles are sub-micron in size.

The relief leaks were slower to seal, initially only one leak was opened at a time, allowing the pressure to build up, once the pressure rose above 20 mbar, a separate relief hole was opened up and the sealing process started again. The sealing process of the relief hole is shown in Figure 81.



*Figure 81: Sealing of the top relief hole in less than 30 minutes* 

Header text



Figure 82: Humidity of system during Test #4.3



After two of the relief holes were sealed and a third was partially sealed the relief section was removed to have the seals photographed from the outside and the inside of the pipe, Figure 83.

#### Header text



*Figure 83: Three of the four 3 mm relief holes were opened and sealed in sequence* 

**Group 4 Test Conclusions** 

# 6.4 Overall Test Conclusions

due to the ease of handling and HSE considerations.

when generating aerosols within the pipe system.

It has however been possible to

tune the system very effectively to seal all of the test leaks and subsequently seal the much larger pressure control leaks.

The main conclusions from the tests can be summarised as follows:

- A number of different dry powder solids have been aerosolised and can be transmitted in pipeline systems, large particle size (> 10 μm) means these solids can fall out of suspension reducing the effective distance of conveyance.
- A number of liquids have been aerosolised and transmitted long distances in pipeline systems. Particle sizes
  < 10 μm aids the suspension of these aerosols making transport over long distances possible.</li>

#### Header text

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- Both systems had a tendency to coat the inner pipe wall and this tendency was greater on the lower half of the pipe wall (due to gravitational effects). The flow regime, in particular speed of flow has a significant effect on this.
- The solid aerosols had a larger particles and therefore dropout was much more significant, the liquid aerosols had much less drop out and can therefore be conveyed over long distances.
- Leak sealing tests have been successfully carried out and characterisation work carried out at the University of Bristol.

. This complements the design

Overall the programme of experiments has been highly encouraging, not only in successfully sealing leaks of different sizes in different locations on the pipe but also in developing an understanding of the mechanism of aerosol generation and evolution such that the leak sealing can be controlled by environmental factors.

The leak sealing ability can be turned on and off which suggests that this could be used to differentiate between leaks and customer supplies. The sealant behaviour can also be tuned by changing the base molecule, for instance a change from s the particles are tuned for optimal sealing.

Changes in the seal if required. seals from undesired locations thus providing

Alternative chemistries can be used and additives applied to produce different seal properties including permanence of seal under a wide range of environmental conditions.

Altogether, the findings provide a large number of opportunities to build on for the detailed engineering stage of the project.

# 7 WP5: Enabling Technologies

It was expected that the use of aerosols would require additional 'enabling technologies' such as mechanisms for generation of aerosols and mechanisms of safely dealing with the aerosols after the sealing process has been carried out. In reality for the work carried out, only a limited amount of equipment has been required. If we examine the stages of the aerosol in turn then potential requirements for enabling technologies can be studied.

It is assumed that the leak sealing aspect of the aerosol is addressed as part of the main engineering; enabling technologies then deal with other aspects of getting the aerosols into the system, getting them out and protecting customer equipment.

# 7.1 Generation

A number of different aerosol generators have been used for the work here. Some of them will be valid for generation in the field, however depending upon the eventual system required the particles may need to be produced in a defined humidity. Options for producing such systems may be spray dryers or similar systems and then mixing the dry aerosol into a gas of known humidity.

It is likely that the generation system will involve a pre-conditioning chamber with a specification dependent upon the aerosol chemistry used.

Generation is expected to occur at a higher pressure than the gas system so this will aid the delivery into the flow system.

# 7.2 Conveyance

During testing, conveyance through the system has been carried out using a simple fan or more simply by the process of aerosol generation producing a high pressure source. This high pressure drives the conveyance of the aerosol through the system. In the field conveyance is likely to be generated by the fact that the riser system will be at a higher pressure than the surrounding air. Conveyance is therefore more likely to be controlled by gas exiting the system to be sealed. This will involve charging the system with aerosol rich gas and then extracting this aerosol from a subsequent point in the system. The enabling technology here would be the extraction unit which should take the aerosolised gas, remove the aerosol particles from the system and the return the clean gas into the network. If the aerosol particles are in stasis with the gas then the recirculated gas can be re-introduced to the system being sealed along with the aerosol to further use the particles and maximise the overall sealing ability.

# 7.3 Termination

This element of technology examines what happens to aerosol particles which are not used to seal leaks and asks how (or if) they are to be taken out of the system. The dispersed nature of the sealing material in an aerosol method makes the termination of sealing activity especially challenging and important. If active aerosol sealant is left in the pipe system there is the risk it will block, partially block, or intermittently block customer outlets. This could cause boilers or hobs in residential properties to extinguish and, until safeguards are activated, continue to flow.

The exact nature of the termination depends upon whether sealing particles are allowed to approach customer supplies.

The ideal system is one which passes through customer supplies without blocking control valves or jets and combusts harmlessly releasing no toxins into the atmosphere. If this is not possible then measures must be put in place to remove particles from the system.

Less attractive systems could involve the isolation of customer supplies during the sealing operation and the subsequent removal of particles prior to re-instating supplies.

Termination can be achieved by one or a combination of the following:

- **Flushing**: ensure flow through all branches and ensure that flow conditions (e.g. medium and velocity) unique to the sealing job are sufficient to remove all free aerosol components.
- Immobilisation: ensure that the components will settle and once settled remain so after known time at defined conditions
- **Degradation**: ensure that the components lose their ability to agglomerate and seal after a known distance
- Combustion: ensure the components of the aerosol are combustible at flame temperature
- **Release**: ensure the components that can survive flame temperature are harmless to people and the environment
- **Collection**: collecting all particles prior to reaching customer supplies.

The exact nature of termination used will depend upon the sealing technology used and will be examined in further detail in subsequent stages of the work.

# 8 Conclusions and Further work

This final chapter examines the outcomes of the project and looks a further work required to take the proposed technology forward to a useable service for leak sealing in the gas network.

# 8.1 Project Conclusions

The overriding conclusion of the project has been that *the concept of using aerosols for leak sealing in a gas environment has been proven*.

The concept of the aerosol has been studied at the single particle level. The change of the particle in relation to interaction with the environment has been observed and modelled resulting in a fundamental understanding of the aerosol behaviour in a gas pipe. By applying this understanding to experiments in the workshop environment is has been possible to control the aerosol particles such that they are optimised for leak sealing. This optimisation was achieved by controlling the aerosol particles. This understanding of

comes from the single particle measurements and this technique can be applied to many different types of aerosol chemistry. The atmosphere in the gas pipeline is then controlled to match the for that particular chemistry.

An additional advantage of using

Once at the seal

the aerosol continues to dry up and subsequently harden to a permanent seal.

Particular outcomes of note are:

- Leaks ranging from 0.3 mm to 3 mm diameter were sealed in different orientations on the pipe wall.
- The aerosol sealing ability is controllable studies carried out.
- The sealing was seen at a position remote from the generation after flowing around a number of 90° bends and pipe sections.
- Very low volumes of sealant were used during the leak sealing tests suggesting an efficient form of sealant delivery.
- Very low levels of deposition were seen on the pipe walls although the sealing system can be changed to favour deposition by increasing the humidity and the particle size.
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These overall conclusions are determined by the end result of the experimentation which in turn has been led by concept tests and subcontracted work carried out during the course of the project. The CFD carried out by Norton Straw informs the interaction between the flow of the gas in the system and the impacts of the particles with the pipe and leak resulting in likelihood of deposition and leak sealing.

The single particle measurements and other aerosol studies carried out by the Bristol Aerosol Research Centre has informed the design and optimisation of the **second second** aerosol. These studies enable the design of the aerosol to be carried out and transferred to different chemistries with other desirable features for the gas network.

This work stands alone in relation to prior art, it is however complementary with that prior art so, although not essential, benefits can be seen in continuing to promote the relationship with Prof Modera at California Davis University and Aeroseal Inc (owners of the Background IP).

Steer will be examining the IP position for leak sealing in the gas network. It may be of value to protect this IP for SGN to maximise the value of this technology going forward.

# 8.2 Further Work

The successful leak sealing tests are very encouraging and suggest that the technique should be investigated further with a view to producing a viable technology for sealing leaks in the low-pressure gas distribution network.

Such a programme of work may include the following elements of work:

- Optimisation of the technique
- Alternative Chemistries and Aerosol Formation
- Protecting Customer Supplies
- Differences in Natural Gas

Each of these elements of work can be further expanded.

#### **Optimisation of the Technique**

It is recommended that a detailed programme of work carried out to explore the abilities of this technique. This should include investigating:

- The effect of particle size
- The effect of
- Maximum and minimum seal sizes
- Behaviour of aerosol at different locations
- Maximum and minimum distance between generation and seal
- Different seal geometries
- The seal pressure capabilities
- The seal makeup and permeability
- Temperature effects

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# Alternative Chemistries and Aerosol Formation

The results of the optimisation experiments can be used to inform an investigation into different glass formers and alternative chemistries. Opportunities can then be proposed to ensure the permanence or longevity of the seal. The exploration of chemistries may include the addition of solids to the sealant solution or additional aerosols promote strength and longevity.

#### **Protecting Customer Supplies**

The need to protect customer equipment must be investigated and the effects of passing the aerosol into representative customer systems, such as boilers and cookers should be investigated. Conditions for the aerosol to pass through should be noted and solutions to protect such equipment should be devised. This work may include:

- Experiments to determine differences between a 'leak' in a pipe and a boiler jet.
- Identification of infrastructure that can be put in place to remove the aerosol prior to reaching customer equipment.
- Identification of infrastructure that can be put in place to render the aerosol harmless by the time it reaches customer supplies.

#### Look at tests in natural gas systems.

As the technology moves closer to field ready an investigation into the behaviour of the technology in representative systems should be undertaken. This may include:

- Study of the theoretical differences between nitrogen and natural gas.
- Testing in representative pipework, such as metal riser systems.

It is recommended that a proposal of work is drawn up for the next stage of this project.

# 8.3 Prior to Next Stage

There are a number of outstanding items requiring clarity prior to progressing with this work. These revolve around the IP situation regarding novel concepts developed in this project and alternative aerosol technologies developed at the University of California. Discussions will be held between Steer and patent lawyers to clarify the position of IP generated in this project with the possibility of protection. Further discussions will be held between Steer and Prof Modera to explore the possibilities of returning to the original aim of collaborative working. These will be undertaken in the coming months. The results of these discussions will feed into the work scope for future stages of this project.