

University of Wales Trinity Saint David

Student - Paul Corry

Student Number - 0750389

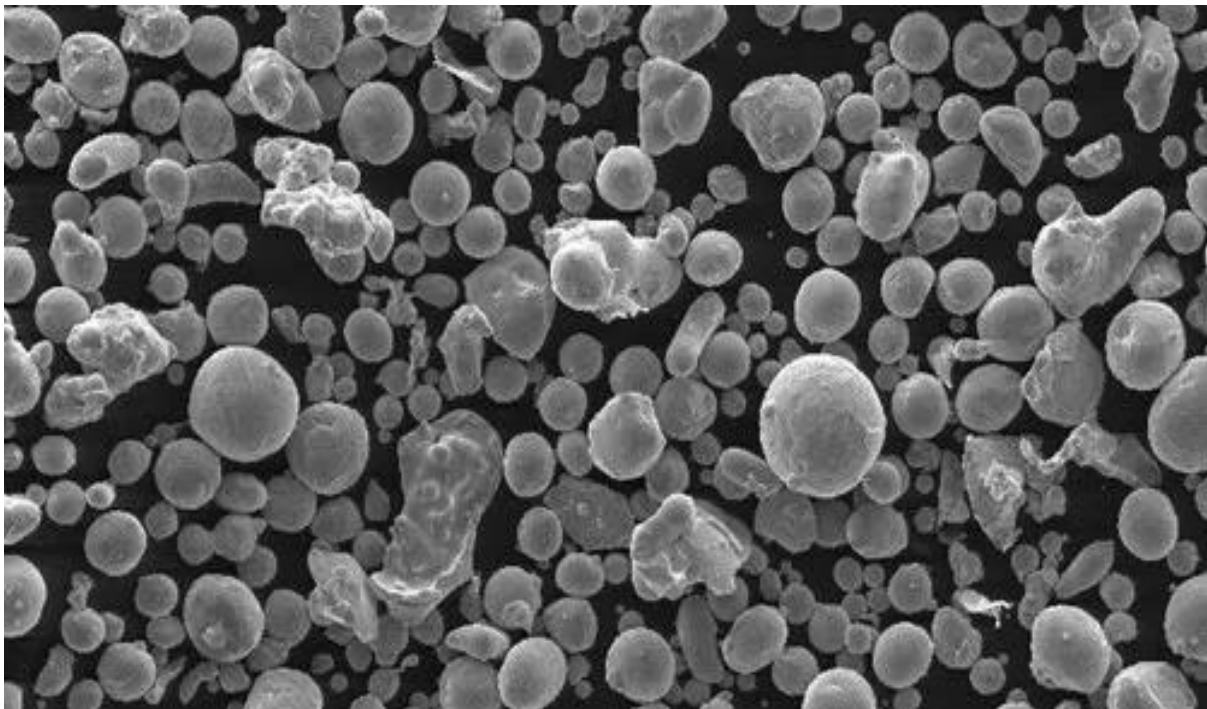
Module Code - ACMA6013

Dissertation Title:

Conversion from Hybrid to Water Atomized
Methodology of a Nickel-Based Hard Surfacing Alloy at
LSN Diffusion Ltd

Word Count-13373

(exclusive of content pages, acknowledgements, abstract and references)



Dissertation Declaration

I hereby declare that this specific thesis/dissertation is the result of my own independent work and investigation. This is an original piece of research whereby; no part has been ever submitted for any type of qualification in any type of educational establishment. The standards of research requirements set out by the University of Wales Trinity Saint David have been adhered to and I can also confirm that this dissertation is a true reflection of my efforts and abilities that I have gained over the last four years of study within the university.

Acknowledgements

I would dearly like to thank all the staff at the University of Wales Trinity Saint David who has given me the opportunity, support and most importantly, the belief of completing my Engineering Materials Degree. Their invaluable guidance and expertise throughout this engineering programme have absolutely instrumental throughout my time at the university. I cannot thank you all enough.

Special mention to Phil Holden, he has been an inspirational to me as a materials lecturer. His wealth of knowledge and how he delivers lectures have been captivating. He truly is a great asset to the university. Thanks also to Kelvin Lake and Peter Charlton who have helped guide me through this dissertation.

I would also like to thank my work colleagues at LSN Diffusion who has supported my studies and given me the valuable time needed to complete my studies whilst working in a very busy environment.

Lastly, I am indebted to my family and friends who have provide me their moral support and encouragement throughout my four-year academic journey. Their unconditional love, understanding and belief in me has no doubt given me the much-needed motivation. For this I am extremely and truly grateful.

Special mention to my wife Julie and daughter Skye for inspiring me on throughout all the highs and lows of my research journey. Without their patience, this degree programme would not have been possible.

I have also gained many great friends both lecturers, and student, on my academic journey.

According to *Hyland, K., (2004)*, an acknowledgment is fairly universal in the dissertation academic practice which, provides an opportunity to convey their gratitude for the personal assistance received while completing their studies.

Abstract

This dissertation ideology was initially set out in an attempt to reduce the manufacturing costs of a particular product that is currently produced by gas atomizationⁱ (nitrogen medium). This material is used in the oil industry as a nickel-based hard-facing metallic powder which, is applied to hard face oil pump shafts via HVOF (high velocity oxy fuel) and also by plasma spray welding application equipment. These types of thermal spray powders exhibit good wear and corrosion properties, therefore, can extend the life of the part/s in service.

Another route available for producing this material would be water atomizationⁱⁱ which, is considered the cheaper option. This dissertation, therefore, aims to investigate this possibility of converting to water atomization with no or minimal detriment to final application.

The energy costs (more so the nitrogen gas) for a typical melt (350 kg) cost around ~£170 per melt for the gas atomized product with a final yield of ~17%. By converting to water atomized, the data in this research clearly shows that this particular material can be produced at an estimated cost of ~£100 per melt and with an increase in final yield to ~24%. This, therefore, virtually halves the energy cost associated with producing this material without compromising on quality of the product. Relative increase in yield is therefore, ~30% which, virtually halves the energy costs of producing this grade which, in turn produces a higher profit for the organization.

In regards, to quality, the data in this dissertation shows that the integrity of the coating has not been compromised. Although the water atomised particles contain more hollows/porosity the grain structure “post” application shows very little difference between both atomizing methodologies.

This research has also led to other materials now being produced at LSN Diffusion by the water atomizing methodology resulting in further financial gains which, have not been mentioned in this dissertation. Orders received will now go through a more thorough process in regards, to contract review to establish whether or not, the water methodology can be applied rather than defaulting to gas.

Contents Page

Dissertation Declaration.....	2
Acknowledgements	3
Abstract.....	4
Contents Page	5
Table of Figures.....	8
1 Introduction.....	11
1.1 Dissertation Background.....	11
1.2 Aims and Objectives	11
1.3 Research Testing Methodology.....	12
2 Literature Review / Further Reading	13
2.1 Literature Review Introduction.....	13
2.2 Literature Review Area of Focus	13
2.3 Literature Review Discussion & Relevant Literature.....	14
2.4 Literature Review Evaluation and Challenges.....	16
3 LSN Diffusion Introduction and Product Overview	18
3.1 LSN Testing Equipment	18
3.2 Thermal Spray Coatings Introduction and Properties	19
3.3 LSN's Manufacturing "Atomizing" Process Map	21
3.4 Atomization Introduction and Principles	22
3.4.1 Hybrid Atomization Methodology.....	25
3.4.2 Water Atomization Methodology.....	27
3.4.3 Dry Gas Atomization Methodology	29
3.5 Typical Atomizing Gas Costs.....	30
3.6 Particle Morphology, Size Distribution and Yield.....	30
3.6.1 Hybrid Atomized Yields.....	33
3.6.2 Water Atomised Yields	33
3.6.3 Mechanical Sieve Shaker	34
3.6.4 Laser Sizing Equipment.....	36
4 Project Scope	38
5 Metallography Sample Examination.....	39
5.1 Metallography Standard Operating Procedure.....	39
5.2 Keyence Digital Microscope.....	41
5.3 Prepared metallography Image Specimens for N-586X4-LC53.....	41

5.3.1	Hybrid v Water >180µm (x100 mag)	42
5.3.2	Hybrid v Water 180/150 µm (x100 mag).....	43
5.3.3	Hybrid v Water 150/125 µm (x100 mag).....	44
5.3.4	Hybrid v Water 125/106 µm (x100 mag).....	45
5.3.5	Hybrid v Water 106/90 µm (x100 mag).....	46
5.3.6	Hybrid v Water 90/75 µm (x100 mag).....	47
5.3.7	Hybrid v Water 75/63 µm (x200 mag).....	48
5.3.8	Hybrid v Water 63/53 µm (x200 mag).....	49
5.3.9	Hybrid v Water 53/45 µm (x200 mag).....	50
5.3.10	Hybrid v Water 45/38 µm (x200 mag)	51
5.3.11	Hybrid v Water 38/20 µm (x200 mag)	52
5.3.12	Hybrid v Water <20 µm (x400 mag).....	53
6	Hollow Particle Findings Evaluation of Both Atomization Methodologies.....	54
6.1	Hollow Particle Findings for Hybrid Atomization.....	54
6.2	Hollow Particle Findings for Water Atomization.....	55
6.3	Hollow Particle Findings Comparison of Both Methodologies	57
6.4	Hollow Particle Findings Comparison 20/53µm Combined PSD Range.....	58
6.5	Hollow Count Possible Explanation in Water Atomized Material.....	60
6.6	Water Atomization Jet Set-up Configuration Investigation	60
6.7	Ishikawa Diagram used for Water Head Set-up Assembly.....	61
6.8	Hollow Count Using New Water Primary Jets	64
6.9	Water Atomization Primary Jets Investigation Conclusion	67
6.10	Hollow Count Conclusion of Both Methodologies	68
7	Yield Considerations for converting from Hybrid to Water for N-586	69
7.1	As Atomized Yield Comparison Data.....	70
7.2	Consistency of Theoretical Yield of Water Atomized Material	72
7.3	Laboratory Evaluation of Finished Screened Material.....	74
8	Product Deposition Comparison of Hybrid V Water Atomised Product.....	75
8.1	Deposit of Hybrid and Water Material	75
8.2	Hybrid Atomised Metallography / Grain Structure and Deposition Porosity Count	76
8.3	Water Atomised Metallography / Grain Structure and Deposition Porosity Count	77
8.4	Deposition Porosity Count Comparison of Hybrid and Water Product	78
8.5	Deposition Conclusion / Findings for Both Atomizing Methodologies.....	78
9	Atomizing Energy Costs Comparison and Evaluation	79
9.1	“Current” Production Costs and Customer Demand for N-586 Material.....	79
9.2	“Current” Production Gas Costs for Hybrid and Water Atomised Material	80

9.3	Hybrid Atomized Material Gas Costs	80
9.4	Water Atomized Material Gas Costs	80
9.5	Gas Costs Comparison and Potential Savings	80
9.6	Current" Production Electricity Costs for Hybrid and Water Atomised Material	81
9.7	Hybrid Atomized Material Electricity Costs	81
9.8	Water Atomized Material Electricity Costs	81
9.9	Electricity Costs Comparison and Potential Savings	82
9.10	Total Production Energy Costs for Both Hybrid and Water Atomized Product.....	82
9.11	Conclusion of Energy Costs of Both Atomizing Mediums.....	82
10	Dissertation Conclusion	83
10.1	Financial Impact to the Organisation.....	84
10.2	Customer Feedback	85
10.3	Dissertation Evaluation / Critique.....	85
11	References	87
12	Endnotes	92

Table of Figures

Figure 2:1. Porosity by Powder Size. Study by Zhong, W et al. (2023).....	15
Figure 3:1. Thermal Spray Principle Process	20
Figure 3:2. Atomizing Processes at LSN Diffusion	21
Figure 3:3. Typical Metallic Atomization Principle	22
Figure 3:4. Induction Melting Furnace Example.	22
Figure 3:5. Gas & Water Atomization Basic Process Principle	23
Figure 3:6. Hybrid Atomizing Assembly	25
Figure 3:7. Gas Head Insert and Ceramic Molten Metal Guiding Nozzle	26
Figure 3:8. Gas Head Insert in Assembly.....	26
Figure 3:9. Water Head Assembly.....	27
Figure 3:10. Water Atomizing Typical Process	28
Figure 3:11. Annular Gas Nozzles used for Dry Gas Atomising	29
Figure 3:12. Hybrid Ni-Based Alloy (x200) ~20/53µm	31
Figure 3:13. Water Ni-Based Alloy (x200) ~20/53µm	31
Figure 3:14. Log-Normal Particle Size Distribution Profile	33
Figure 3:15. Rotap Mechanical Sieve Shaker	35
Figure 3:16. Lazer Sizer used to Determine PSD	37
Figure 3:17. Particles Scatter via the Laser Beam	37
Figure 5:1. Metallography Polished Samples Hybrid & Water Atomized Particles	39
Figure 5:2. Metallography Numbered Samples Both Hybrid & Water.....	40
Figure 5:3. Standard Operating Procedure for Hollow Count	40
Figure 5:4. Keyence digital Microscope	41
Figure 5:5. Hybrid Polished Particles >180µm (Ref-H1 x100 mag).....	42
Figure 5:6. Water Polished Particles >180µm (Ref-W1 x100 mag)	42
Figure 5:7. Hybrid Polished Particles 180/150µm (Ref-H2 x100 mag)	43
Figure 5:8. Water Polished Particles 180/150µm (Ref-W2 x100 mag).....	43
Figure 5:9. Hybrid Polished Particles 150/125µm (Ref-H3 x100 mag)	44
Figure 5:10. Water Polished Particles 150/125µm (Ref-W3 x100 mag).....	44
Figure 5:11. Hybrid Polished Particles 125/106µm (Ref-H4 x100 mag)	45
Figure 5:12. Water Polished Particles 125/106µm (Ref-W4 x100 mag).....	45
Figure 5:13. Hybrid Polished Particles 106/90µm (Ref-H5 x100 mag)	46
Figure 5:14. Water Polished Particles 106/90µm (Ref-W5 x100 mag).....	46

Figure 5:15. Hybrid Polished Particles 90/75 μ m (Ref-H6 x100 mag)	47
Figure 5:16. Water Polished Particles 90/75 μ m (Ref-W6 x100 mag)	47
Figure 5:17. Hybrid Polished Particles 75/63 μ m (Ref-H7 x200 mag)	48
Figure 5:18. Water Polished Particles 75/63 μ m (Ref-W7 x200 mag)	48
Figure 5:19. Hybrid Polished Particles 63/53 μ m (Ref-H8 x200 mag)	49
Figure 5:20. Water Polished Particles 63/53 μ m (Ref-W8 x200 mag)	49
Figure 5:21. Hybrid Polished Particles 53/45 μ m (Ref-H9 x200 mag)	50
Figure 5:22. Water Polished Particles 53/45 μ m (Ref-W9 x200 mag)	50
Figure 5:23. Hybrid Polished Particles 45/38 μ m (Ref-H10 x200 mag)	51
Figure 5:24. Water Polished Particles 45/38 μ m (Ref-W10 x200 mag)	51
Figure 5:25. Hybrid Polished Particles 38/20 μ m (Ref-H11 x200 mag)	52
Figure 5:26. Water Polished Particles 38/20 μ m (Ref-W11 x200 mag)	52
Figure 5:27. Hybrid Polished Particles <20 μ m (Ref-H12 x400 mag)	53
Figure 5:28. Water Polished Particles <20 μ m (Ref-W12 x400 mag)	53
Figure 6:1. Graph % of Hollows per Particle Size Fraction	57
Figure 6:2. Hybrid Particles 53/20 μ m (x300 mag)	59
Figure 6:3. Water Particles 53/20 μ m (x300 mag)	59
Figure 6:4. Ishikawa "Cause & effect" Used for a Water Head Set-up Assembly	61
Figure 6:5. "Used" Primary Water Jet (x100 mag)	62
Figure 6:6. Used Primary Water Jet	62
Figure 6:7. "New" Primary Water Jet (x100 mag)	63
Figure 6:8. New Primary Water Jet	63
Figure 6:9. Water Assembly Set-up Actual Drawing	64
Figure 6:10. Graph % of Hollows per Particle Size Fraction for All Tests	66
Figure 6:11. Particle Size Distribution Plots of both Water Lot-79168 & 79759 (as atomized)	67
Figure 7:1. Difference in % Between Hybrid & Water (53/20 μ)	71
Figure 7:2. Plots of both lots: Hybrid 79256 & Water 79168 (as atomized)	71
Figure 7:3. Water Atomized Material Consistency (53/20 μ)	72
Figure 7:4. Water Atomized Old v New Theoretical Values % (53/20 μ)	73
Figure 8:1. Deposition of Hybrid and Water Atomized Material	75
Figure 8:2. Metallography Grain Structure Image Deposition (x1000) Hybrid Material	76
Figure 8:3. Metallography Grain Structure Image Deposition (x1000) Hybrid Material	76
Figure 8:4. Metallography Grain Structure Image Deposition (x1000) Water Material	77
Figure 8:5. Metallography Grain Structure Image Deposition (x1000) Water Material	77

Table of Data

Table 1-1. Typical Chemical Composition of N-586X4.....	12
Table 3-1. Atomising Gas (Nitrogen) Costs.....	30
Table 6-1. Hybrid Atomized Hollow Count % by PSD Fraction	54
Table 6-2. Hybrid Summary Hollow Count 20/53µm Test Count 1 & 2 Average.....	55
Table 6-3. Water Atomized Hollow Count % by PSD Fraction	56
Table 6-4. Water Summary Hollow Count 20/53µm Test Count 1 & 2 Average	56
Table 6-5. Conclusion of Hollows Spheres by Atomizing Methodology.....	58
Table 6-6. Water Atomized Hollow Count Using "NEW" Primary Jets % by PSD Fraction	65
Table 6-7. Comparison of % Hollows of Water Atomized Product (Original set-up v New Jets).....	66
Table 6-8. Summary Hollow % Count of Atomizing Methodologies	68
Table 6-9. Current Water Atomization Parameters	68
Table 7-1. "As Atomised" Particle Size Distribution of both lots: Hybrid 79256 & Water 79168.....	70
Table 7-2. Water Atomized "As Atomised" Theoretical Analysis Taken from Several Lots.....	72
Table 7-3. "As Atomised" PSD Comparison of Water Lots 79168 & 79759 (old v new jets)	73
Table 7-4. Final Laboratory Evaluation Hybrid v Water Atomization	74
Table 9-1. Atomizing Parameter & Demand Requirements (Hybrid v Water)	79
Table 9-2. Atomizing Methodology Nitrogen Gas Costs (Hybrid v Water)	80
Table 9-3. Atomizing Methodology Electricity Power Costs (Hybrid v Water).....	81
Table 9-4. Total Atomizing Energy & Annual Melt Requirement Costs (Hybrid v Water)	82

1 Introduction

1.1 Dissertation Background

From late 2022 energy costsⁱⁱⁱ have risen sharply within the organisation which, has the knock-on effect on the costs of the product being produced. For example, both electricity and nitrogen costs have increased by nearly fifty percent which, in some cases cannot be passed onto the customer. Therefore, much emphasis is now on to reduce internal costs of the organisation to produce products will lower manufacturing costs to stay competitive in the marketplace. According to *Smith, R. and Hawkins, B., (2004)*, continuing to be reduce operational costs are vital to for maintaining profitability within any organization. Nitrogen alone makes up approximately ~80% of energy consumed for the atomization of alloy powders, however, recently in 2023 costs have increased further still by nearly forty percent therefore, the total costs associated with purchasing nitrogen has virtually doubled.

1.2 Aims and Objectives

This dissertation, therefore, is aimed to explore and consider an alternative atomizing methodology such as, water Atomization in order to save and reduce gas costs by producing such a general hard-facing self-fluxing nickel-based alloys with little or no detriment to customer application and of the material quality deposition.

A particular product which, is currently manufactured by the hybrid route^{iv} (gas atomised using nitrogen and collected in water) is applied by customers in the North America. The material is applied as a general hard-surfacing to coating to oil-drill shafts which, is predominantly in the oil exploration industry. This nickel-based alloy is an extremely wear resistant material which also has a hardness of ~60HRc. Due to the boron and silicon ratio, this material has a fairly low melting fusing temperature, therefore, produces a smooth clean surface post the application process which, aids the final grinding/finishing process of the pump shafts^v. However, for not too dissimilar applications, water atomized material is often used. Typical Nominal Composition for this alloy can be shown in Table 1-1. Material was selected for this project due to its low production yield of ~17%.

Table 1-1. Typical Chemical Composition of N-586X4

Boron	Carbon	Chromium	Iron	Silicon	Nickel
3.2%	0.7%	16.0%	4.0%	4.5%	Balance

1.3 Research Testing Methodology

Research methodology according to *Schutt (1996)*, can be used as a way of assessing the validity of particular research. Research can be of three main types depending on its intended purpose such as explanatory, descriptive, and evaluative. These research techniques can be classified into methodologies as qualitative and also quantitative. Quantitative research uses actual data in numerical form. It can often be utilised to find patterns and make predictions by analysing the data gathered whereby, qualitative research is the opposite which, using non-numerical data which cannot generalise to the wider population such as case studies for example although, for this dissertation quantitative will be predominantly used, secondary sources such as journals, books and web sites will also be evaluated/critiqued. As a means of justifying the data/results of this study, a clear precise methodology is vital for the research process (*Khalid, K., et al., 2012*).

As stated by *Feather, D., (2012)*, research methodology is a way of clarifying how a researcher proposes to carry out their particular research. It follows a systematic and logical plan to resolve a problem and details a researcher's approach to ensure reliable and valid results which, addresses its aims and objectives.

According to *Disman, D., Ali, M. and Barliana, M.S., (2017)*, research testing methodology used in this particular dissertation which, is common in undergraduate work includes some of the following components such as data collection methods, sample testing strategies, data analysis and its reliability.

Test methodologies that will be used in this dissertation will comprise of:

- Particle size distribution - using mechanical sieves and laser sizers.
- Hollows count/evaluation - metallography resins and polishing equipment.
- Particle sphericity evaluation - using a high powered Keyence microscope.
- Deposition metallography - welding application to evaluate deposition grain structure.

2 Literature Review / Further Reading

2.1 Literature Review Introduction

As stated by *Burns, (2000)*, a literature review can provide a stimulus for summarising relevant research. It aims to review points made of recent knowledge on a particular theme which, is being investigated therefore, the ultimate target is to bring the reader up to date on the research. It is important however, that a literature review is characterized by logical flows of ideas in combination with an unbiased view of the previous work that has previously been undertaken. As well as identifying good ideas and key concepts, a literature review can also help to evade mistakes which previous researchers have made (*Randolph, J., 2019*).

2.2 Literature Review Area of Focus

This literature review examines differences between hollow particles of water and that of hybrid atomized methodology. According to *Anderson, O. et al (2000)*, hollow spheres have now gained attention due to their physical properties and the ramifications on application in thermal coatings. Thermal spray coatings are predominantly used for enhancing in regards, to their durability and performance in many industries. As stated by *Ren, Z. et al. (2023)*, atomizing method is of utmost importance depending on the customers' requirements and of their application.

This literature review focuses on explaining the many factors that influence the quality of coatings. The quality of metallic powders ultimately influences particular properties such as porosity and microstructure for example, therefore, this literature review attempts to highlight and examine the differences between both methodologies as physical properties of powders of both atomizing methodologies can have significant effect upon the materials sphericity, density and also its flowability which, may have an impact on the final manufactured part. This, therefore, plays an important part in the decision-making process of both methodologies (*Stankevich, A., 2017*).

2.3 Literature Review Discussion & Relevant Literature

As stated by *Pengjun, F. et al. (2018)*, materials produced by gas atomization involves fast solidification from the nitrogen gas which, results in the formation of finer metallic spherical powder that can characterized by having a superior packing density and good flowability^{vi} due to its near spherical morphology and its smooth surface. Although, on the other hand as mentioned by *Asgarian, A. et al. (2020)*, water atomized can exhibit more irregular particles which, can affect their flowability thus, inferior to gas atomised powders. These particles however, again due to fast cooling rates may result in finer microstructures, therefore, improves strength which, can be beneficial in some types of industries such as sintering for example.

It is also mentioned further by *Yenwiset, S. and Yenwiset, T. (2016)* and *Persson, F., (2021)*, that the water atomized more irregular particulates are formed due water turbulence and also from the splattering which, is associated with all types of metallic powders manufactured by the water process. This, therefore, differs from material produced using gas as its atomizing medium whereby according to *Zhong, W., et I. (2023)*, the powder particles are more generally smoother and uniform in shape.

It is argued by *Sypeck, D.J. et al, (1998)*, however, that the production of hollow spheres with water can present challenges due to the turbulent nature of this particular process. Although, water produced material can exhibit an improved martensite formation which, aids the coatings wear resistance and its toughness. This can be improved further by “tweaking” the chemical composition which, in turn, subtly improves its surface morphology. On the other hand, as mentioned by *Sobolev, V. et al. (1995)*, hybrid (gas) atomised material due to its uniform particle size distribution have reduced trapped porosity which, can contribute to its higher tensile strength and such other improved physical characteristics than compared to that of the water atomized product.

A comparative study of a nickel-based super alloy powder was conducted by *Zhong, W, et al, (2023)* in regards, to porosity of the powder particles. This study however, concentrated on the distribution of porosity of particle sizes of between 60µm and 180µm. However, the material in this instance was manufactured using “argon” as the atomizing medium. Examination of the powder particles was conducted using a “Zeiss” electron microscope is as shown in Figure 2:1. Interestingly, on evaluation, the results showed that the porosity/hollows were evenly distributed at the edge and the centre of

the metallic particles however, it was noted that the particles over 120 μm contained the biggest pore size and less or no obvious porosity on the powder particles that were less than 60 μm . Porosity within the particles reached ~90% (nine in every ten particles) above 180 μm . It was therefore, determined that porosity was reduced the finer the material become. Explanations include the shrinkage during solidification and also argon being enclosed when spheroidized.

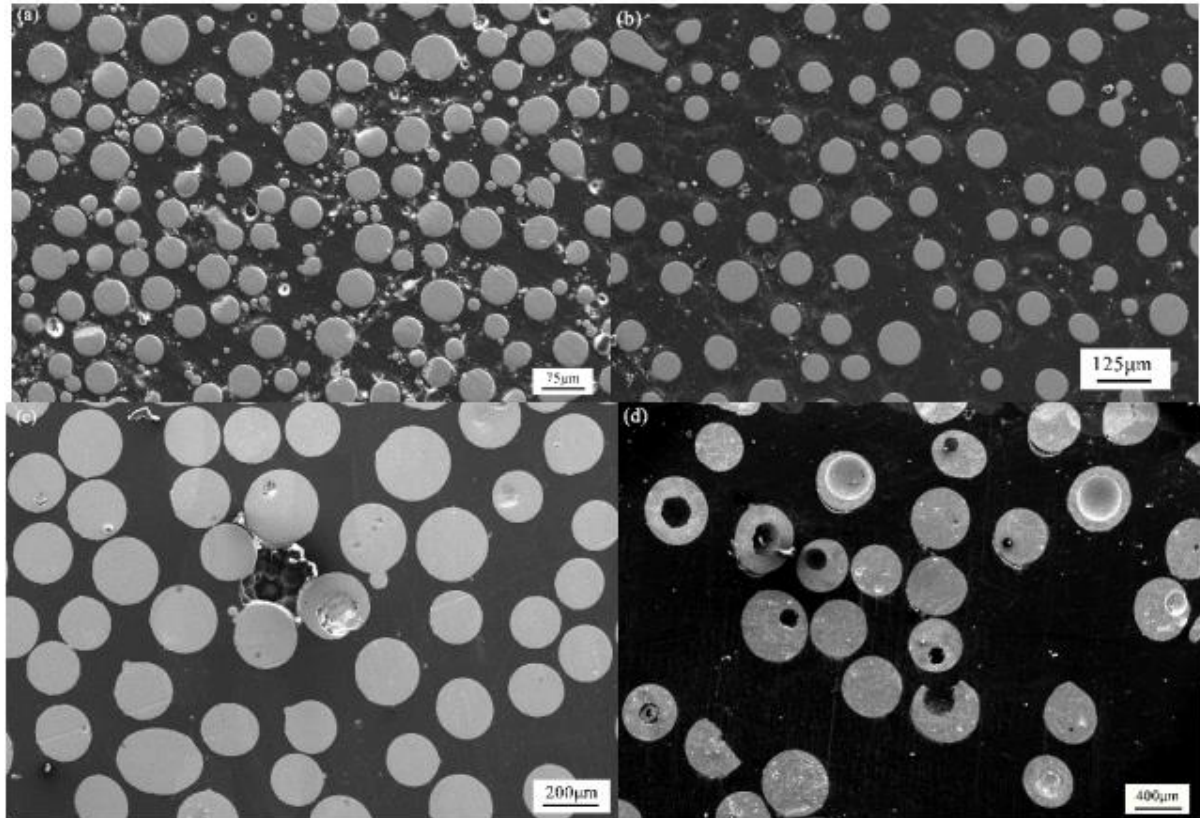


Figure 2:1. Porosity by Powder Size. Study by Zhong, W et al. (2023)

As mentioned by Yenwiset, S. and Yenwiset, T., (2016), molten metal temperature, water pressure/flow rate and also the jet configuration are considered important factors which indeed, affects the particle size distribution as well the powder particle shape. This particular study investigated the shape and size of the metallic particulates by using two different types of nozzle diameter sizes which, dictates the molten metal to water ratio. All other atomizing conditions remained to same. Both sets of results yielded very different as atomized particle size distributions. The small size yielded finer powder which, was measured by microtrac equipment. However, it is important to note although, that this study was conducted on copper material.

Another type of study which, was conducted by *Fedina, T. et al., (2020)*, focused on the differences of both water and of gas atomized powders. However, this was carried out on low alloyed iron-based material (steels). Focus in this instance was the difference in densities between both methodologies and their performance in laser additive manufacturing as the material packing densities for this particular application is an important factor.

The results of this research showed that the water atomized material was approximately fifteen percent less dense than the gas atomised material. High-speed imaging technology was used to observe both types of material in regards, to its behaviour and powder movement under a process such as laser irradiation. It was also observed/noted that the water atomized material had less particle entrainment. A higher oxygen content in the water atomized material explained why the water atomized material had more splattering as well as melt pool instabilities than the gas atomized material.

2.4 Literature Review Evaluation and Challenges

From this literature review, It can be therefore, assumed that both atomization methodologies for the production of metallic powders can have different physical and metallurgical characteristics although, both methodologies have varying atomizing parameters which, can be used such as pressures and nozzle size for example to subtly their physical characteristics such as size and shape to create metallic powders with tailored properties.

Evidence in this literature review indicates that hybrid (gas) atomized powders can exhibit consistent and more spherical particles than material produced by water although, as mentioned by *Lagutkin, S. (2004)*, can be more expensive to produce. Additionally, it is important to mention that gas atomization methodology may not be totally suitable for all types of nickel-based alloys. For example, customers may dictate a particular Hall Cup^{vii} physical flow rate in their specification for application purposes. Water atomized material contain more irregular particles, which, in turn slows down the physical flow of material.

New Analytical techniques such as x-ray imaging and laser diffraction can now play a major role in explaining these relationships of powder characteristics. Atomizing parameters can therefore, optimized based upon these results (*Gray, J. and Tillack, G.R., 2001*).

However, further research and developments are needed to further optimize both water and hybrid material in regards, to their physical properties and morphology. By developing more understanding of the relationship of hollow spheres and their performance researchers, can explain the full potential in the thermal spray coating industry.

Depending on customers specific application and criteria, material both water and gas can have advantages and disadvantages. However, despite the differences as detailed in this review, both methods of atomized material can have their respective roles in numerous industries. To produce material, which is constant in regards, to quality and also its reproducibility remains a key challenge. Although, what is not detailed here is that the sphericity of both methodology changes in size.

As previously mentioned, only the application itself and the materials intended function can dictate which, type of atomizing medium would be best suited. Therefore, the choice of water or gas atomized powders does depend on desired metallurgical properties and most importantly, cost considerations.

Water atomized material is classified as the cheaper option whereby, applications are not so heavily reliant upon the materials particle shape and surface of the finished coating especially where cost efficiency of the application is paramount. Gas produced material on the other hand generally produces more uniform and also more dense spherical particles which, therefore, exhibits increased packing densities and improved surface finish. This is vital for the higher end application industries such as additive manufacturing for example (*Kassym, K. and Perveen, A., 2020*). Therefore, the selection of water or gas atomized material is a decision based purely on the balance between the required properties and it's intended application.

3 LSN Diffusion Introduction and Product Overview

LSN Diffusion is a leading specialist manufacturer of atomized metallic thermal spray powders which, can be supplied as either Nickel, Iron or Cobalt based. These powders can be used for general hard surfacing, high temperature brazing, hiping and laser additive applications, and come with a spectrum of particle size range distributions and chemical compositions depending upon customers final requirement or application. LSN customer base include industries such as: Glass, Dental, Automotive, pharmaceutical, and Aerospace for example, therefore, all material produced at LSN Diffusion must be of high quality.

Nickel-based alloys both for hard-facing and brazing applications make-up the majority of the alloys produced at LSN Diffusion, (approximately 80%). These alloys are assembled (similar to a cake recipe) then melted using induction coil furnace technology and atomised in a medium of either nitrogen or water. Material is then sieved into various fractions dependent upon the application requirement and sent to laboratory for final inspection and certification.

3.1 LSN Testing Equipment

Typical laboratory test consists of the following:

- Chemical analysis using ICP (wet method).
- Gas analysis using sophisticated analysers which, measures C, S, O and N.
- Screen analysis using mechanical test sieves combined with laser sizers.
- Deposition of powder using various welding applications (PTA etc).
- Hardness testing of powder deposits.
- Hall Cup flow and density checks.
- General morphology / sphericity examination.

The values obtained from all these various inspection tests are all validated and evaluated which eventually determine the release of the material to customers.

3.2 Thermal Spray Coatings Introduction and Properties

According to *Nouri, A. & Sola, A., (2019)*, Thermal spray material can be a method for protecting a base-material or substrate and can also be used to improve the surface properties depending on the material intended application.

Feedstock of metallic powder is heated using gasses in application which, are accelerated and propelled by high speed onto the intended prepared substrate in a melted or even semi-melted state through various spray-weld type applicators. The powders chemical composition, particle size distribution and also its morphology can determine the thermal spray coating microstructure and may also affect its thermo-mechanical performance.

As mentioned by *Amin, S. and Panchal, H. (2016)*, thermal spray metallic powders are widely used in industry for multiple applications which, can have the following common-most thermal spray properties:

Metallic thermal spray powders exhibit enhanced wear resistant coatings thus, making them most suitable for protecting the surface whereby, abrasion or indeed friction is a particular concern. Such coatings increase the longevity of the component. Thermal coatings are also essential in maintaining structural integrity in high-temperature environments in industries such as in aerospace and gas turbines.

Another important use of thermal spray coatings is for corrosion protection when the structures, or components are used in hostile or corrosive/acidic environments. The coating, therefore, acts as a barrier which, protects the substrate again, extends the lifespan of the part (*Qinyang Li. et, al 2019*).

Material can be deposited to restore worn or damaged parts. An example of this is in the glass industry whereby, thermal spray material is commonly used to repair bottle making moulds damaged by the hostile environment of molten glass.

From these mentioned examples, thermal spray metallic powder therefore, offers multipurpose solutions in many industries seeking enhanced performance and also the durability of particular components or even structures therefore, according to *Fauchais, Et, Al., (2014)*, thermal spray powders play a crucial role in wide ranging applications in many organizations.

Thermal spraying, therefore, offers cost-effective solutions for the enhancement of surface properties by using precision control in regards, to chemical composition versatility and thickness compared to other traditional surface protection methodologies such as, electroplating (*Talib, R.J., & Saad, S., et, al, 2003*). Metallic powders are now becoming indispensable in industry by offering all kinds of surface coating solutions. The principle of the thermal spray process can be seen in Figure 3:1.

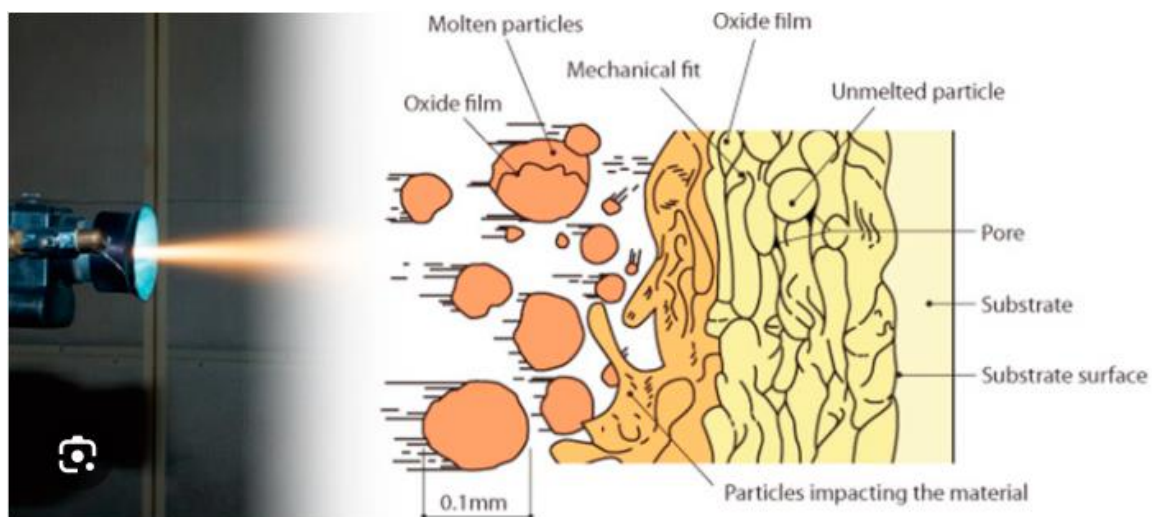


Figure 3:1. Thermal Spray Principle Process

3.3 LSN's Manufacturing "Atomizing" Process Map

Melt charges or heats are calculated in regards, to its required chemical composition and assembled using either raw element, revert material (previous oversize or undersize material) or both which, is then melted to a homogeneous state using sophisticated induction type furnaces. This molten metal is then converted into powder by a choice of three atomizing process manufacturing routes as depicted in Figure 3:2. Material is then processed by classification (dust removal) and screening to the required particle size distribution as per specification. Finished screened material is mechanically blended into a homogenous mix and subjected to laboratory tests such as ICP (inter coupled plasma) to determine its final chemical analysis composition and other physical testing such as particle size distribution, laser sizers for example. The finished sample will also be subjected to deposition testing using various application welding equipment. Hardness values can then be taken from these deposits along with the chemical analysis for final certification purposes.

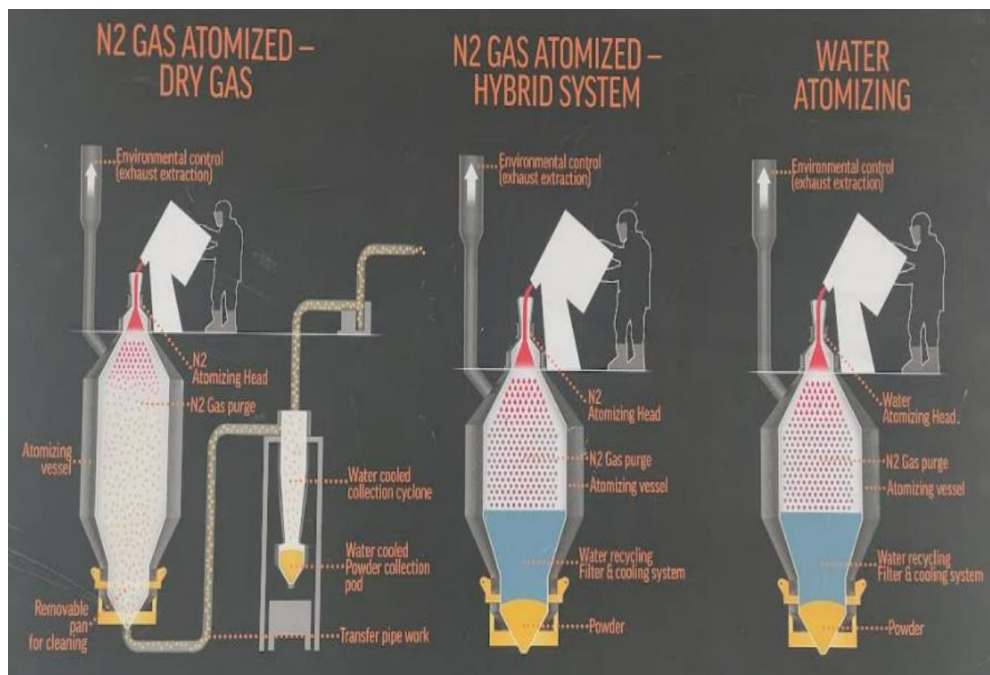


Figure 3:2. Atomizing Processes at LSN Diffusion

3.4 Atomization Introduction and Principles

Powder atomization is a process whereby a bulk liquid (molten metal), is broken up into small powder droplets by high pressure jet of gas (nitrogen) or by water depending on customers application or requires specification of the manufactured finished product. In other words, atomization can be referred to as separating something into finer particles (*Lagutkin, S. et al, 2004*), as shown in Figure 3:3.

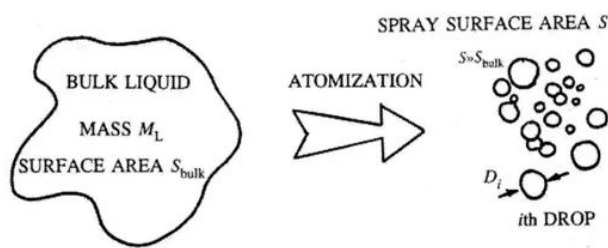


Figure 3:3. Typical Metallic Atomization Principle

Ceramic nozzles which, are zirconium-based, guide the molten alloy stream from the induction furnace as in Figure 3:4 and into the atomizing chambers which consists of various types of atomizing head configuration assemblies whereby the atomizing medium is controlled by parameters such as, pressures or nozzle diameter size. The objective of these atomizing parameters is to optimize metal to gas/water ratio to achieve predetermined particle size distributions so maximum screening yield can be obtained (*Emami, M. 2013*).

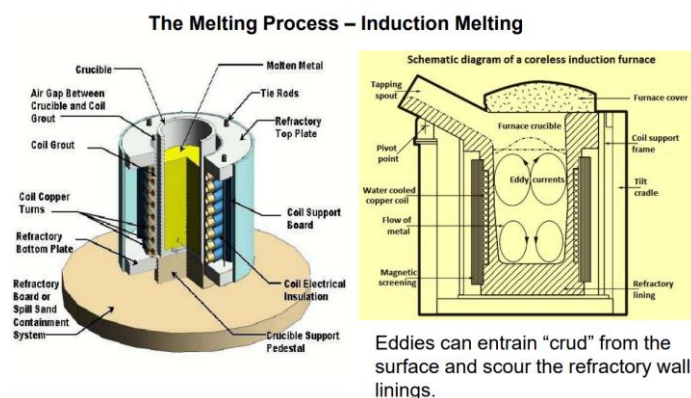


Figure 3:4. Induction Melting Furnace Example.

As shown in Figure 3:5, to break-up the melt into droplets, the molten melt stream from the high velocity of gas or water jets in the form of kinetic energy. High pressure/slow melt flow in regards, to gas atomizing produces finer powder due to the gas to metal ratio. Likewise with water atomization, whereby higher pressure/slower molten metal flow also produces finer powder. By these variable parameters, material particle size distribution can be tailored to maximise yield.

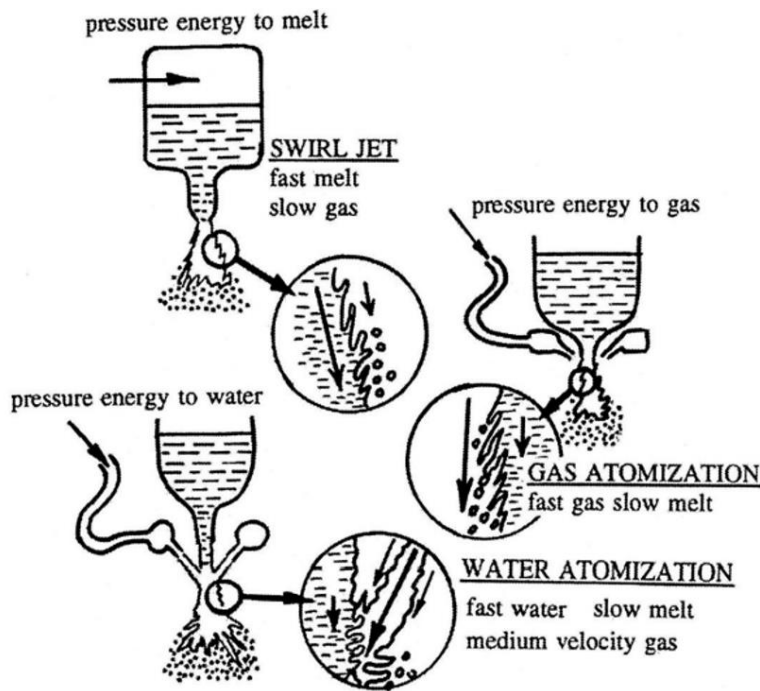


Figure 3:5. Gas & Water Atomization Basic Process Principle

As stated by Lagutkin, S. et al, (2004), atomizing can, therefore, be defined as a two-step process which, firstly shears the molten liquid and secondly the sheared liquid (droplets) freezes or solidifies. However, the rate of freezing can depend upon the surface tension of the molten metal and the temperature of the molten droplets which, is dependent on the chemical composition of the alloy being produced. The transfer of molten metal to the pre-heated tundish must be however considered as heat can be lost in this process as well as the atomising itself. The tundish is “topped-up” with molten metal from the induction furnace. The difference/varying of temperatures in this process can affect the cooling and morphology of the particle, therefore, melt parameters/work instructions are created for melt consistency simplicity. (Jiang, X., Siamas, G. A. et al, 2010).

The following are all types of atomizing methodologies available at LSN Diffusion:

1. Dry gas atomization - Nitrogen atomized dry collect.

Material is atomized using nitrogen as the atomizing medium. The atomised metallic powder is dry collected as in a sealed pod under atmosphere.

2. Hybrid atomization - Nitrogen atomised wet collect.

Atomized as per the dry-gas route. However, the material is wet collected (in water) and subsequently dried using rotary nitrogen swept driers.

3. Water atomization - Water atomised wet collect.

Material is atomized using water as the atomizing medium. The powder as per the hybrid system is collected in water and dried using rotary nitrogen swept driers.

All three atomizing methodologies have varying costs associated with them. Selection of methodology normally depending on the customers final application. Although, on many occasions default atomizing using the hybrid methodology is selected and added to the material specification if the customers application requirements are not fully known or sales are through a third-party distributor when ordering general hard-facing nickel-based alloys. Third-party distributors are used as they can serve as intermediaries between end users and the manufacturers. They can also tap into established markets and offer logistical support whereby the manufacturer cannot easily achieve.

3.4.1 Hybrid Atomization Methodology

Hybrid atomising inert gases such as nitrogen is used to break up/shear the stream of molten metal. The liquid metal stream is fragmented by the rapid expansion of the nitrogen from a typical gas assembly head as shown in Figure 3:6. The flight of these particle cool, freeze and are collected in water in the bottom of the atomising system. According to *Lavernia, et al., (1992)*, the particle size distribution can be controlled by the volume and pressure of the nitrogen gas and also from opening diameter of the atomizing ceramic nozzle. Depending on the alloy composition make-up, several gas head assemblies can be used (*Fritsching, U. and Uhlenwinkel, V., 2012*).



Figure 3:6. Hybrid Atomizing Assembly

A typical nozzle and jet configuration is as shown in Figure 3:7, guides the molten metal from the melt's tundish and into the jet/nozzle assembly whereby is intercepted by the nitrogen gas stream. As mentioned by *Le, T. and Henein, H., (1996)*, Geometry and position of this set-up can affect the particle size distribution of the atomized material.



Figure 3:7. Gas Head Insert and Ceramic Molten Metal Guiding Nozzle

The jet and nozzle configuration sits within the atomizing head assembly itself shown in Figure 3:8. The molten metal is transferred from the induction furnace into the jet/nozzle assembly via the tundish which, sits above this head assembly.



Figure 3:8. Gas Head Insert in Assembly

Post atomizing, material is dried using nitrogen swept rotary driers. Water is driven-off as steam and replaced by nitrogen to prevent oxidation. Once dried, the material is discharged into powder containers for further powder sizing.

3.4.2 Water Atomization Methodology

This type of atomization is a process when controlled jets of water are sprayed onto a molten metal stream. The molten stream is broken into smaller granules which, then solidifies. There are a few limitations to this process such as particle shape and composition purity for example, higher Oxygen content and lower apparent or tap densities. However, in particular applications such as hvof^{viii} or plasma spraying^{ix}, Oxygen and percentages of non-spherical particles is not an issue although, as water atomized particles become finer, the sphericity of the particles becomes not too dissimilar from equivalent hybrid atomized material. A benefit of water atomization is the faster remelt due to the solidification of the particle. Water atomizing from a commercial perspective is a method of producing material at the lowest possible cost is the most commercial and economically used process in order, to produce the largest tonnage of atomized powders. However, as mentioned, it does have its limitations. (Asgarian, A., Tang, Z., et al., 2020).

The water atomizing head assembly comprises of various water jet nozzles as seen typically as in Figure 3:9. Primary and secondary nozzle which can be selected depending on the particular particle size distribution required (Ghassemieh, E., et al, 2006).



Figure 3:9. Water Head Assembly

Similar to that of the hybrid method, the molten metal is poured from the bulk induction furnace into a tundish whereby a ceramic nozzle guides the molten metal into the centre of these four primary and secondary water jets which, intercept the molten stream thus converting this stream into metallic droplets.

Again, similar to the water atomized, material is dried using nitrogen swept rotary driers whereby excess water is removed. Material is then subjected to classification (dust removal) and sieving.

The water head set-up configuration and also the primary water jets must be aligned perfectly to prevent splash back and molten metal build-up. This procedure is paramount as material produced by off-centre can result in excessive oversize which reduces the overall yield of the material. Figure 3:10 shows a simplistic water atomization process.

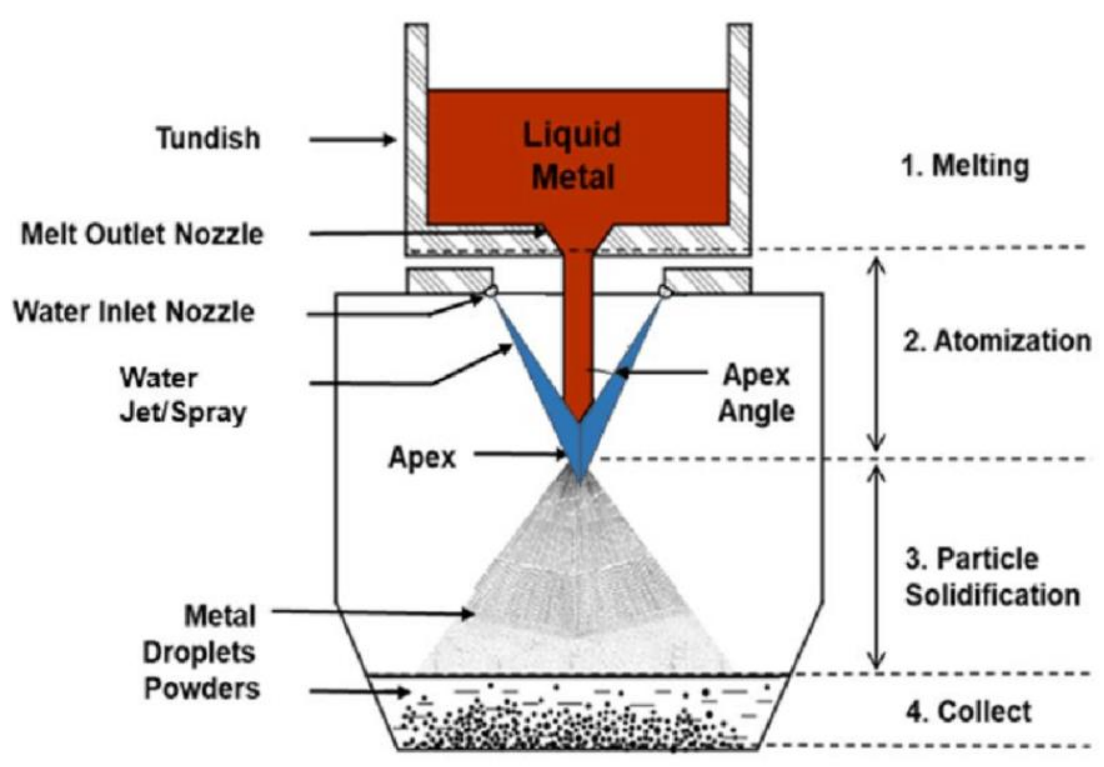


Figure 3:10. Water Atomizing Typical Process

3.4.3 Dry Gas Atomization Methodology

Material atomized by the dry gas system route are classified as “exotic alloys” whereby oxidation via the drying process can be an issue (alloys such as Inconel, Satellites etc). The dry gas atomizer is similar to that of the hybrid atomiser however, the material is dry collected using nitrogen swept cyclone/cooling vessels therefore material produced have a lower oxygen content therefore, no conventional rotary drying required.

Due to the material selected for the dry-gas system, oxygen monitors are placed throughout the dry-gas system as these types of alloys can be susceptible to oxidation at lower temperatures. However, alloys produced using this system tend to be lower in quantities (~10% of all alloys produced) Although, larger atomizing systems are currently being looked at for “potential” larger quantity orders.

The nozzles as shown in Figure 3:11, are of a annular design which, have a higher gas flow to metal ratio therefore, the atomized particles depending on composition tends to be more spherical in shape (Wang, G., et al, 2023), however, with a lower oxygen content than the water atomized material.

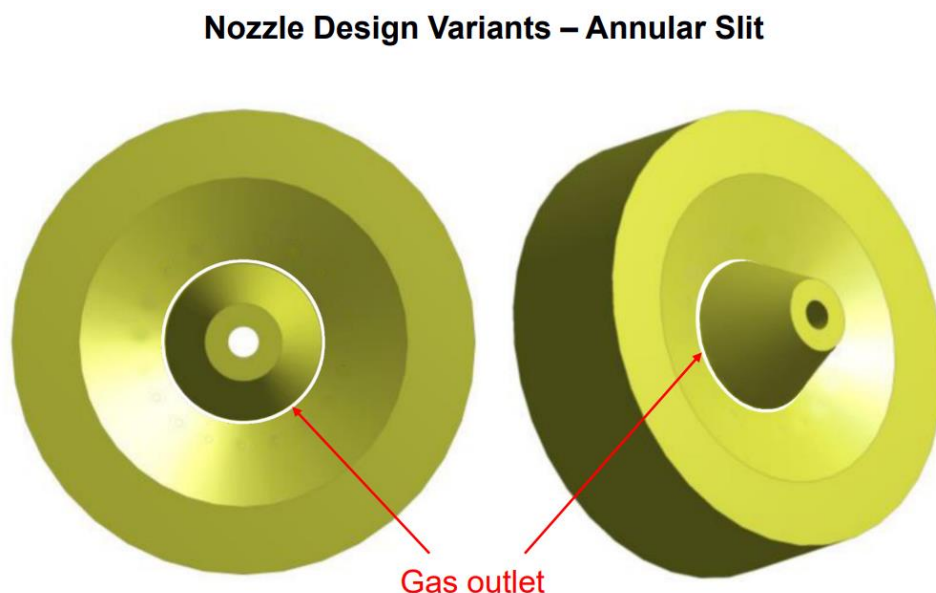


Figure 3:11. Annular Gas Nozzles used for Dry Gas Atomising

3.5 Typical Atomizing Gas Costs

The three atomizing methodologies have varying atomizing costs as illustrated in Table 3-1. Energy costs are calculated from the volume of gas and the kwh of electricity used per melt which, are used for accounting purposes for determining the selling cost of the material. These costs are, however, subject to change depending upon unit energy surcharges imposed on the organisation by the supplier.

The most cost-effective option to produce atomized powder would be the water route however, as mentioned there are limitations. This is followed by the hybrid route which, using nitrogen although, various head designs can be used to reduce this cost subject to the specification of the manufactured product. The dry gas system ultimately used the most gas consumption which, is used for “exotic” and other metallic powders whereby oxidation may be an issue therefore, this system has additional nitrogen swept post atomization collection vessels.

Table 3-1. Atomising Gas (Nitrogen) Costs

Water Atomization Wet Collect	~£100 per 350kg Melt
Hybrid Gas Atomization Water Collect	~£170 per 350kg Melt
Dry Gas Atomization Dry Collect	~£200 per 350kg Melt

Material produced by water methodology as discussed in chapter 3.5, is a more cost-effective method to manufacture atomised powder due to water being used rather than nitrogen in the atomized chamber. For most general hard-facing thermal spray nickel-based powders, water atomized material is the most widely used in this particular type of industry. The major factor which, determines this choice of atomizing methodology is based on price alone. The glass industry is typical of this whereby, many hundreds of tonnes of material is sold in this minimal margin competing business.

3.6 Particle Morphology, Size Distribution and Yield

The three atomizing manufacturing process routes which, are currently available typically have subtle differences in regards, to the morphology and sphericity of the atomized particle although, chemical composition can also determine shape due to the freezing rates, surface tension for example.

However, as mentioned hybrid atomized particles are generally more spherical than that of water atomised particles. Hybrid atomized particles also are denser than water (they suffer from less hollows) which, can potentially cause application quality issues. Dry gas atomized are more varied due to varied chemical compositions although, this particle type of atomized material can suffer from secondary satelliting^x as the finer particles float around the atomizing system and re-attaches to the larger particles prior their solidification or freezing temperature. Both water and hybrid material have a not to dissimilar morphology from an “as atomized” state as a whole. Although coarser hybrid material does contain more spherical particles. As atomised is defined in this instance as “material atomised and dried but with no physical splitting of particles” (un-sieved/screened). However, as mentioned, as the material from both atomising methodologies becomes finer, ($<63\mu$), the particles are virtually indistinguishable from each other. This can be observed in Figure 3:12 and Figure 3:13.

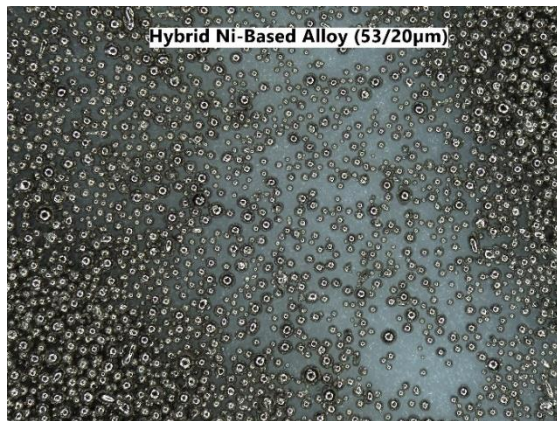


Figure 3:12. Hybrid Ni-Based Alloy (x200) $\sim 20/53\mu\text{m}$

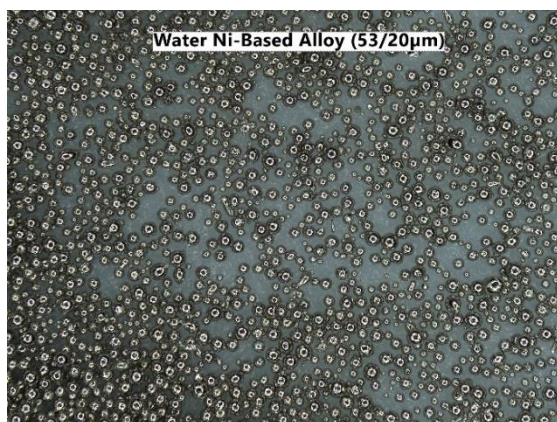


Figure 3:13. Water Ni-Based Alloy (x200) $\sim 20/53\mu\text{m}$

There can be a couple of ways in which these finer particles can be identified from each other:

1. Oxygen Content

Producing material via water medium, particles “depending on chemical composition” have a higher oxygen content than hybrid by nearly 30% on a like for like nickel-based alloy. Although, in many cases, higher oxygen values have little or no detriment to final application.

2. Hollow Spheres

It has been suggested by previous research by *Mostafaei, A., Hilla, C., et al., (2018)*, that water atomised material have slightly more particles with “fresh air/gas” trapped within them. These particles are therefore less dense therefore application parameters may need to be amended to consider the spray-efficiency. It is assumed that these hollow spheres can all burn-up in the flame depending upon the size of the void within the particle which, may cause issues in the final overlay coating such as micro-porosity.

To check for hollow particles, material can be mounted in a resin which holds the powder in place. Once the resin sets, the samples are polished using metallography equipment. Using high powered digital technology images can be observed and these so-called hollows can be quantified from batch to batch. This method is used periodically as a QC tool to ensure the water head set-up is optimised.

In particle science, the size of the frozen metallic droplets normally follows a log-normal type of distribution as shown in Figure 3:14. In most atomizing processes, the happening of a log-normal distribution can explain why a small particle is just as likely as a larger particle to be broken-up by the velocity of the atomizing medium.

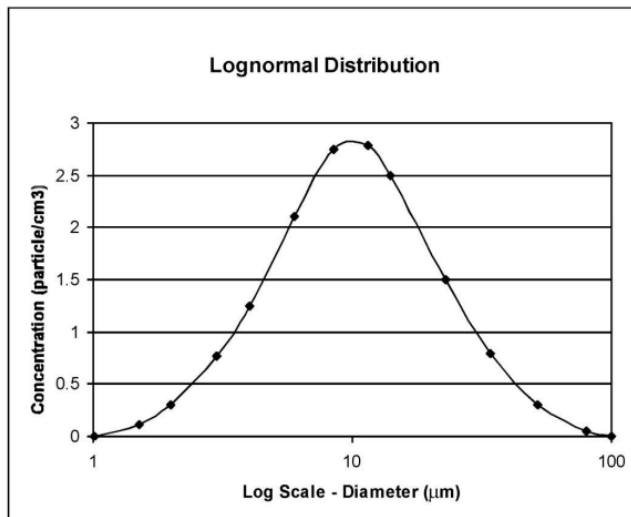


Figure 3:14. Log-Normal Particle Size Distribution Profile

Yield can be considered one of the most important factors when producing metallic powders. Various atomising methodologies as mentioned in this project produces varying yield even on the same product. By using optimized atomising parameters on each atomizing methodology, yield predictions can be made. Depending on final particle size distribution (PSD) for the specific application, varying these atomizing parameters can maximise final yield.

3.6.1 Hybrid Atomized Yields

Hybrid Atomized material using the “free flow” head configuration generally produces coarser powder than that of water atomized. This, therefore, is the preferred method for producing “spray-fuse” type of material which, require particles to be above $\sim 45\mu$. Another advantage is that as clearly demonstrated in this study, produces coarser solid spheres which, is beneficial for this spray-fuse process although, hybrid atomization consumes expensive nitrogen gas.

3.6.2 Water Atomised Yields

Water atomization especially using the 25/8 water head configuration produces more yield of powder towards the finer end ($< 53\mu$) which, for reasons why this is the preferred atomizing methodology when producing “fuse-weld” (puddle weld) type material. Material for this fuse-weld application is generally

below $\sim 75\mu\text{m}$. Increased hollow spheres/particles do not normally cause any ramifications to this particular application process.

According to (*Pasupathy, M. et al, 2016*), calculations can be made to predict the medium size of the atomized powder however, only to a particular alloy family and the atomiser used. Quantitative measurements of the as-atomized material (using laser sizers and test sieves) can determine finished yield. Other quantitative measurements of test such as tap and apparent density^{xi} and also qualitative observations such as the particle morphology^{xii} can confirm the importance of the heat transfer rate or solidification time. Correlations from all these types of data aid maximum yield efficiency and quality when producing metal alloy powders. There are currently three atomizing routes available to produce Nickel hard facing material: Each methodology has varying as atomized particle size distributions. From this collected data yield/s can be predicted (*Persson, F., et, al, 2012*).

Metallic powders manufactured at LSN can be measured by either sieve or laser technology depending upon the finished screened/sieved particle size requirement and also by the request of the customer for certification purposes. Often, values obtained both testing methodologies are combined to produce a single particle size distribution. Both methodologies have advantages and disadvantages.

3.6.3 Mechanical Sieve Shaker

A mechanical sieve shaker such as, a Rotap, is a particle analysis device which is, used in many kinds of industries in order, to test the percentages of particles in a sample in different aperture fractions. This operates by a uniform rotary motion that “shakes” particles down through a series of various micron/mesh sized test sieves in a stack. The analysis is calculated by the percentage of particles which, are retained on the test sieves after the operational time of the test.

To use this method, a representative/homogeneous sample that is poured into the top larger aperture test sieve. This test starts when the shaker is turned-on for a specific period of time. Once completed the material which, is left on each test sieve is weighed as a percentage. This represents the particle size distribution analysis. See Figure 3:15

Advantages of using the sieve shaker:

- Simple method which, is inexpensive.
- Consistency of results.
- Mimics the “actual” production screening of the material tested.

Disadvantages of using the sieve shaker:

- Not suitable for finer particles $<50\mu\text{m}$ (38μ for metallic powders).
- Blinding or pegging of apertures (finer sieves) resulting in inaccurate results.
- Impossible to measure when using cohesive or agglomerated materials.
- Does not account for particle shape.



Figure 3:15. Rotap Mechanical Sieve Shaker

3.6.4 Laser Sizing Equipment

Laser sizer/analysers as such as laser for example, are also used to measure the size of particles within a homogenous sample. The percentages of particles are measured by the angle of the light which, is scattered by the particulates/particles as they pass through the laser beam. Laser sizing can be of dry or wet methodology depending upon customer requirements.

Particles that are large scatter the light at the small angles which, are relative to the beam whereas, the smaller particles scatter light at the larger angles. What determines the size of the particles is the analysis of the angular intensity scattering data.

Advantages of using laser sizers:

- Quick to measure agglomerated material.
- Can measure particulates in a liquid matrix or suspension.
- Accurately measure of fine particles between $\sim 25\mu\text{m}$ $\sim 0.1\mu\text{m}$.

Disadvantages of using laser sizers:

- Takes shape into account (measure of the larger size of particle)
- Costly equipment.
- Differences in values obtained when using different laser sizer models.

A typical laser sizer used at LSN can be seen in Figure 3:16

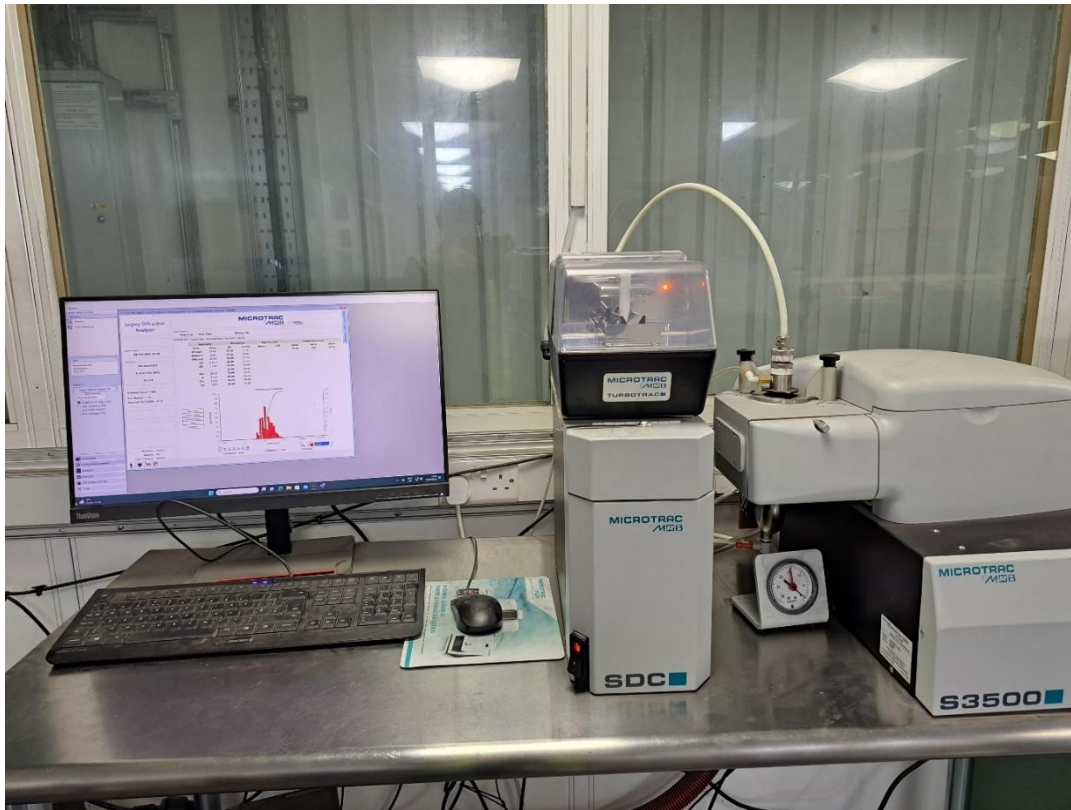


Figure 3:16. Lazer Sizer used to Determine PSD

This specific laser determines particle size by diffraction as per Figure 3:17

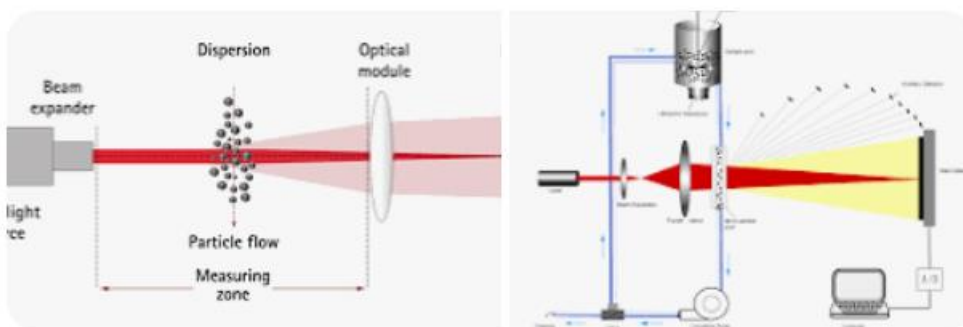


Figure 3:17. Particles Scatter via the Laser Beam

4 Project Scope

From the literature review research done in regards, to examining material at different fractions for hollows spheres in both water and hybrid material, it appears that in both atomized methodologies particles of $\sim 53\mu\text{m}$ and below contain not to dissimilar numbers of hollows. This led to the idea that this particular product could possibly be produced using the cheaper water atomizing manufacturing route as the fine metallic water atomized particles have a similar morphology, apparent and tap density to that of the hybrid methodology. The sphericity of the metallic particles is important for physical flow via customers application. To enhance physical flow, material often dosed with fumed silica which, act as a lubricant between the particles and improve the slip plane.

5 Metallography Sample Examination

5.1 Metallography Standard Operating Procedure

In regards, to the hollow count, “as atomized” material of both hybrid and water were screened at various fractions using calibrated laboratory test sieves on a vibratory shaker which, is primarily used to separate material into particular sized fractions. Each section was mounted in an acrylic resin and examined under the Keyence microscope after metallographic polishing for hollow count evaluation. Metallographic polishing is an important procedure in metallography whereby it's vital to ensure that the microstructure is visible. Sample must be fairly scratch free and is achieved by using grinding papers and polishing down with fine micron sized cloth (*Das, S., 1999*).

Using a powder riffler^{xiii}, representative samples of both hybrid and water material were taken and split into 12 fractional micron size parts using calibrated laboratory test sieves and given a reference number of H (for hybrid) and W (for water). All powder samples were mixed and mounted with an acrylic type of material in order, for the metallic particles to stand proud so they can, therefore, can be polished using metallography equipment for examination under a highly powder microscope. As stated by *Lawley, A. and Murphy, T.F., (2003)*, metallography is a laboratory technique used to study grain structure etc. Polished samples as shown in Figure 5:1 and Figure 5:2, were subjected to a hollow count evaluation. Each sample was prepared in the same way in regards, to the time and quality so a comparison can be made between each sample.



Figure 5:1. Metallography Polished Samples Hybrid & Water Atomized Particles



Figure 5:2. Metallography Numbered Samples Both Hybrid & Water

After polishing, the samples of both hybrid and water particles were then examined closely under a Keyence high powder digital microscope so a hollow count could, be conducted figure 21 to fig 44). Photographical images as seen were used to physically count the number of hollow particles so a calculation could be made to determine their percentage against the solid metallic particles present. The count was performed twice so an average could be used. This particular methodology chosen could be considered subjective although, after consideration only methodology chosen appears logical as only a comparison between hybrid and water is required.

A typical procedure for this particular practise can be seen in Figure 5:3 . The ideology of this type of procedure is to ensure consistency and repeatability of such testing.

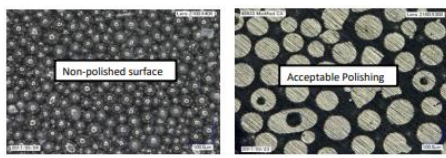
LSN			STANDARD WORK INSTRUCTION		Personal Protective Equipment (PPE)									
Porosity Count Inspection														
Product :	All Powder Products	Original Date:	04.05.2017		Safety Glasses	Safety Footwear	Ear Protection	Respirator	Face Shield	Gloves	Met Particulate Dust Mask	Work Wear	Hard Hat	Bump Cap
Area :	Laboratory	Revision Date:	N/A		Mandatory	Available Not Mandatory	Available Not Mandatory	Available Not Mandatory	Available Not Mandatory	Mandatory	Mandatory	Mandatory	Available Not Mandatory	Available Not Mandatory
Process :	Porosity Count	Issued By:	KA											
Machine:	Keyence Digital Microscope	WI No.:	WI132											
	Struers LaboPol-5 Grinder Polisher	Revision	0		To determine the amount of hollow particles % present in a powder sample, through inspection of a mounted cross-sectioned sample of resin-hardened powder									
	Kemet KEP Resin Hardener Kit													
No.	DESCRIPTION				VISUAL AID									
1	Ensure all equipment is clean and ready for use. Obtain a rubber mould and carefully scoop blended powder into mould. Try to keep amount of powder as thin as possible but covering the entire bottom surface of the mould. Do not tap or allow powder to segregate in mould. If segregation occurs, repeat the entire process.													
2	Fill the graduated mixing cylinder with 2X unit volume of Kemet KEP Resin. Then carefully add X units volume of KEP Kemet Hardener, so that the final ratio by volume of Resin:Hardener is 2:1. Mix well. For every sample it is recommended X = 3 ml.													
3	Fill the rubber mould and powder with the mixture of resin-hardener and place over the hotplate for 30-60 minutes (or 24 hours in atmospheric conditions). Use this time to clean up mounting workspace before any spilled resin hardens.													
4	Once satisfied with the level of hardness from the resin, extract the hardened sample from the rubber mould. Use the letter engraver to label the back of the sample with WO and Alloy Code													
5	Attach the desired grit paper over the GritFix and attach to Struers LaboPol-5 Platen. Ensure the platen is clean and grit paper is free from scratches.													
6	Open the water valve over the platen and polish the sample. Follow Met-Prep Flow Charts / Instruction Sheet to determine correct polishing steps. Recommended RPM 100-200.													
7	Dry the sample under airblower immediately after polishing and examine under microscope. If the polish level is acceptable (see visual aid images) then proceed to image taking. If image quality is not acceptable, adjust grit size and polish again.													
8	Choose comfortable magnification size and image sample under microscope. ~ 8 images are required so long as the images have a combined minimum total of 800 particles (1000 if D50<75µm). Save in designated folder on L: drive													
9	Turn off Microscope Light Bulb. Then, Using software or printed sheets of the images, count the total number of solid particles and the total number of hollow particles													
10	Input the Data into Hollow Database and calculate % porosity				Calculation of Porosity: $\% \text{ Porosity} = \text{Number of Hollow Particles} / (\text{Total Number of Hollow} + \text{Total Number of Solids})$									
11	Ensure all equipment and workspaces are left clean and tidy.													

Figure 5:3. Standard Operating Procedure for Hollow Count

5.2 Keyence Digital Microscope

Samples produced in the organisation are examined under a high powered digital microscope Keyence VHX-700 as shown in Figure 5:4 . This 4K highly powered high-powered microscope it is capable of capturing super high-resolution images as well as producing measurements when examining all types of material.

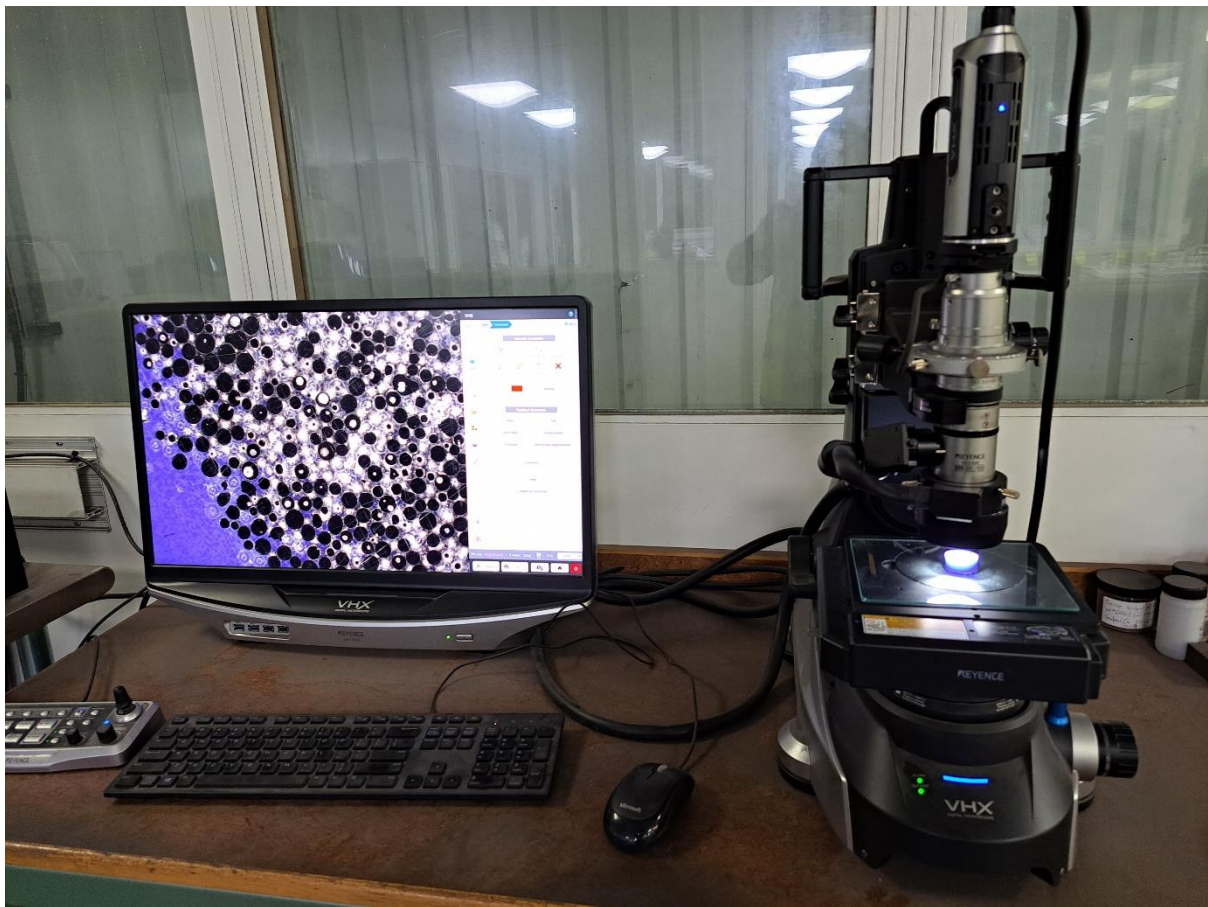


Figure 5:4. Keyence digital Microscope

5.3 Prepared metallography Image Specimens for N-586X4-LC53

Images of these specific fractions of both hybrid and water atomized are shown in Figure 5:5 to Figure 5:28.

5.3.1 Hybrid v Water >180 μ m (x100 mag)

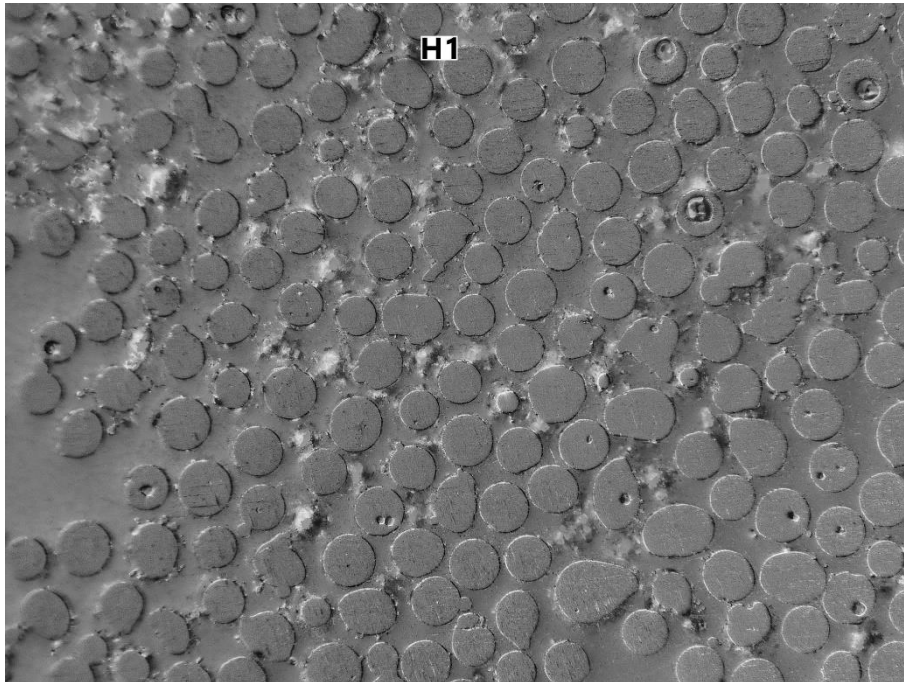


Figure 5:5. Hybrid Polished Particles >180 μ m (Ref-H1 x100 mag)

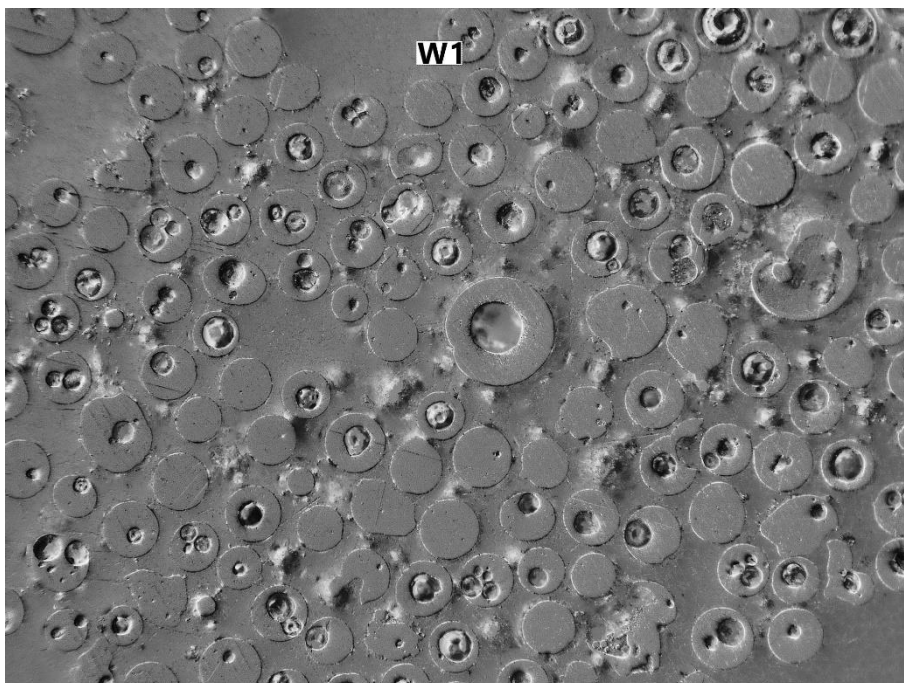


Figure 5:6. Water Polished Particles >180 μ m (Ref-W1 x100 mag)

5.3.2 Hybrid v Water 180/150 μm (x100 mag)

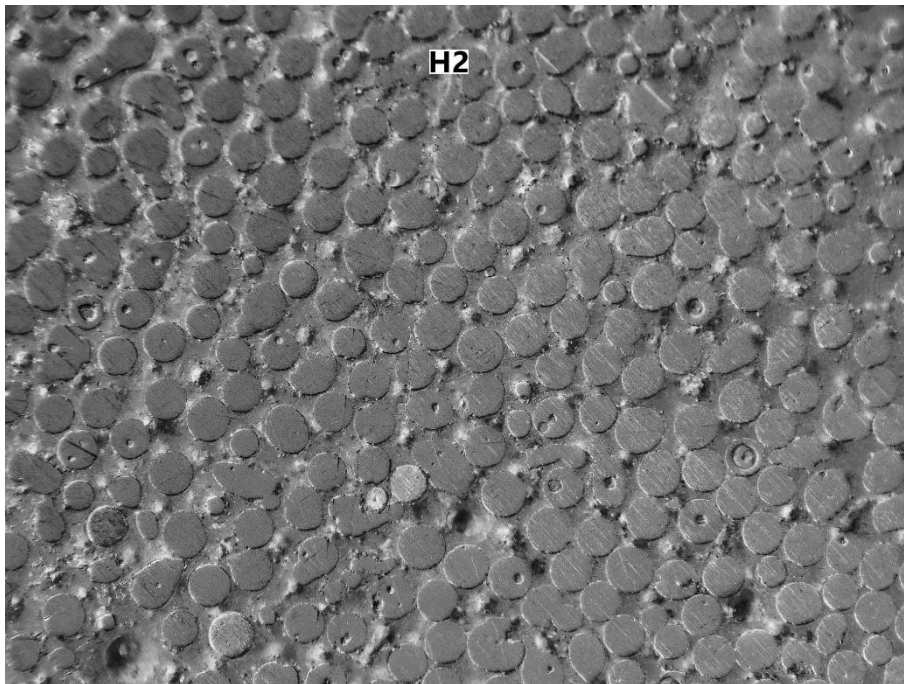


Figure 5:7. Hybrid Polished Particles 180/150 μm (Ref-H2 x100 mag)

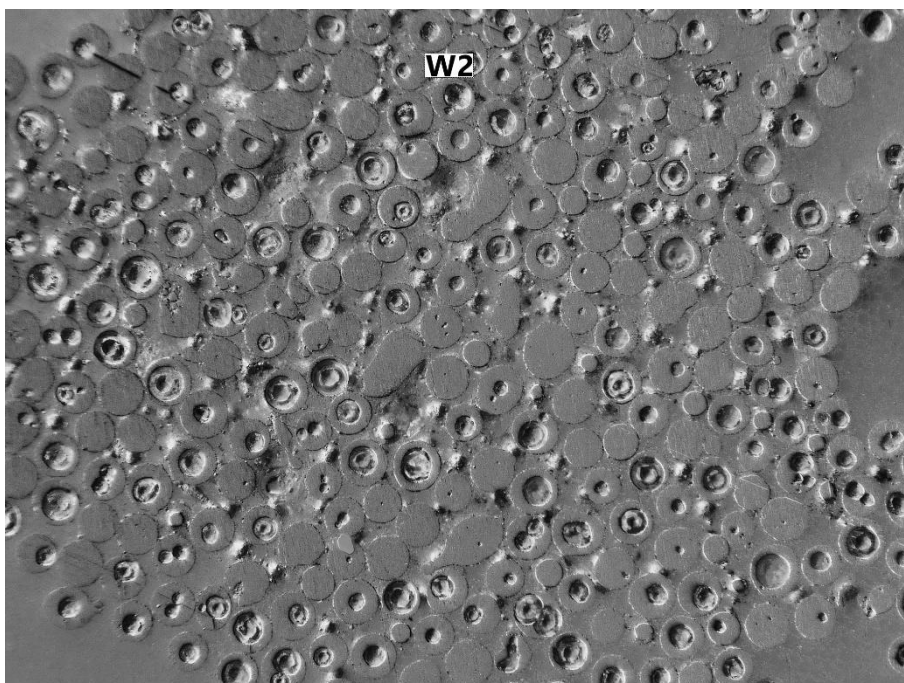


Figure 5:8. Water Polished Particles 180/150 μm (Ref-W2 x100 mag)

5.3.3 Hybrid v Water 150/125 μm (x100 mag)

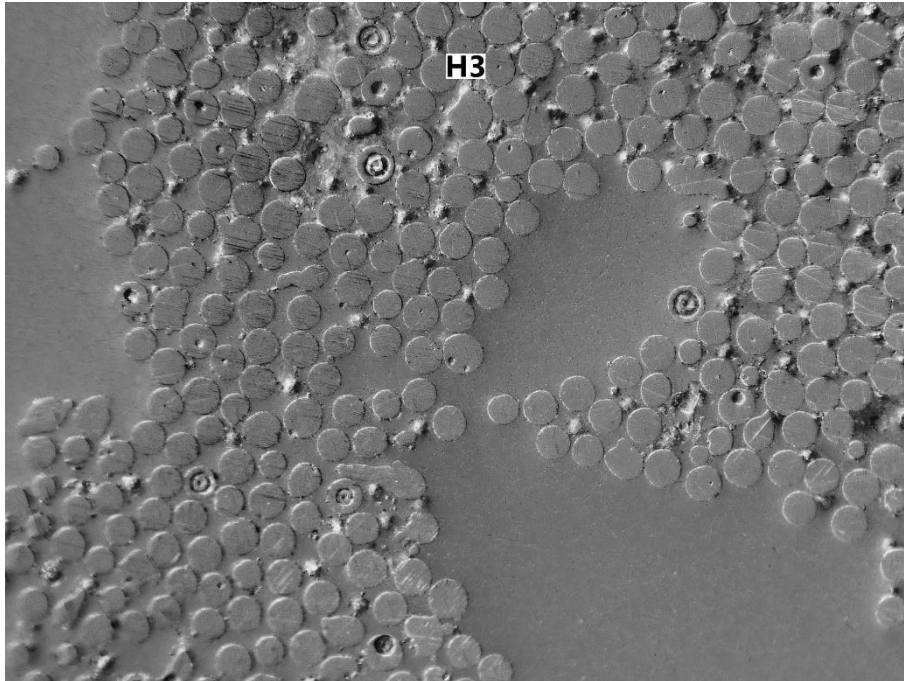


Figure 5:9. Hybrid Polished Particles 150/125 μm (Ref-H3 x100 mag)

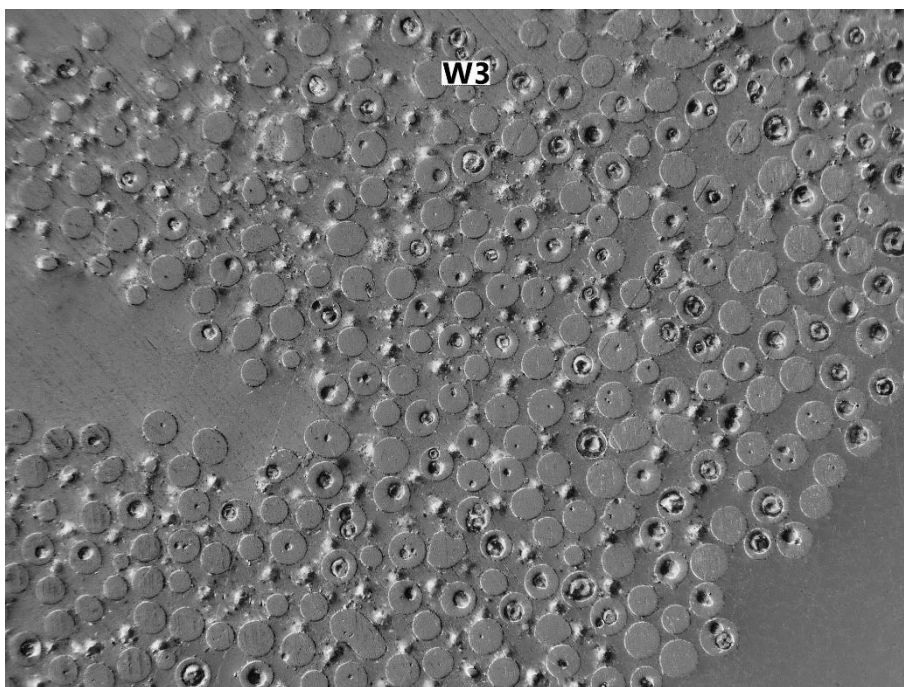


Figure 5:10. Water Polished Particles 150/125 μm (Ref-W3 x100 mag)

5.3.4 Hybrid v Water 125/106 μm (x100 mag)

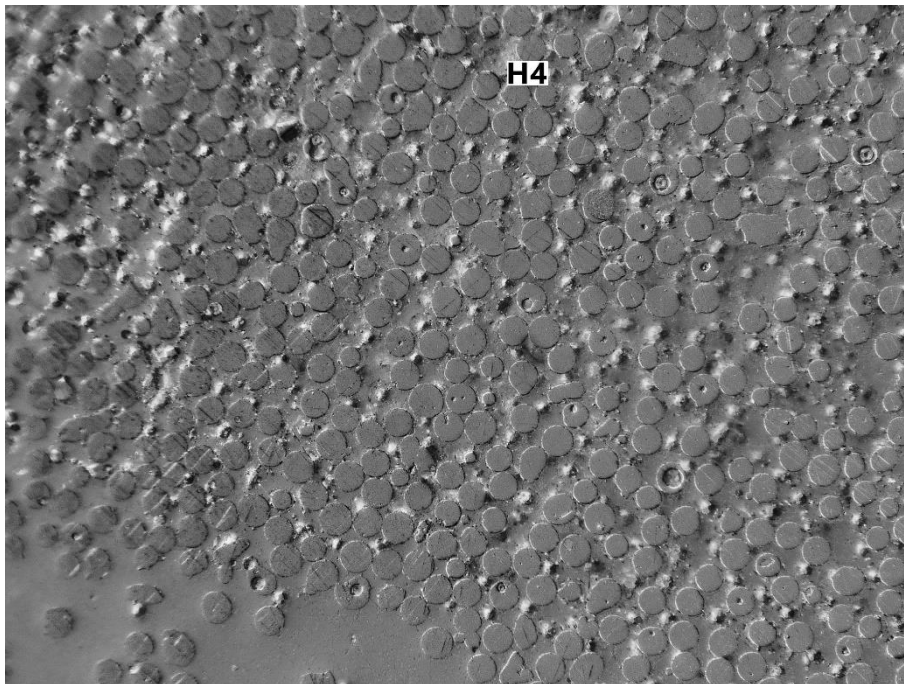


Figure 5:11. Hybrid Polished Particles 125/106 μm (Ref-H4 x100 mag)

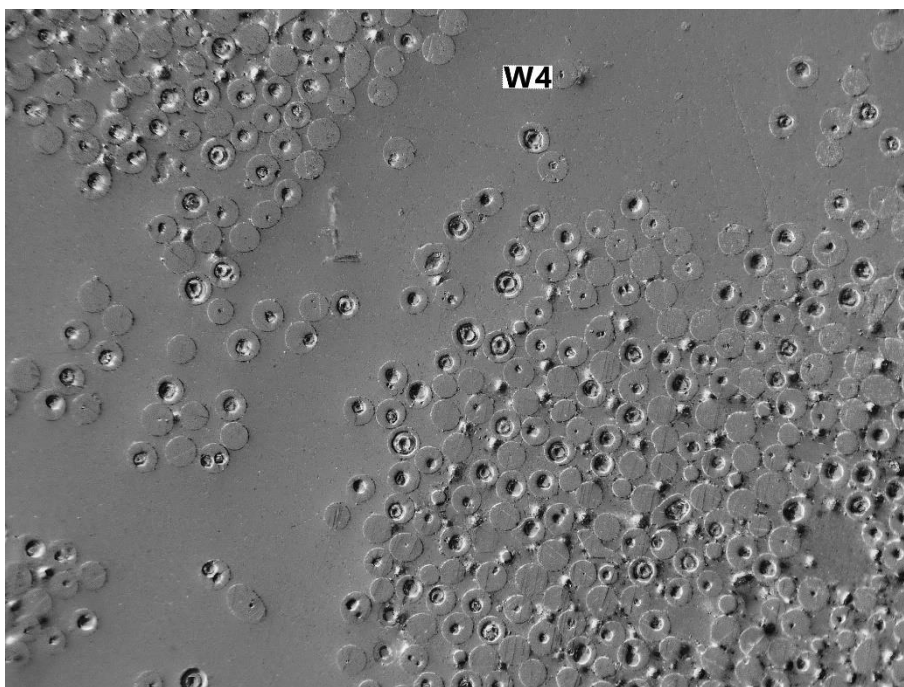


Figure 5:12. Water Polished Particles 125/106 μm (Ref-W4 x100 mag)

5.3.5 Hybrid v Water 106/90 μm (x100 mag)

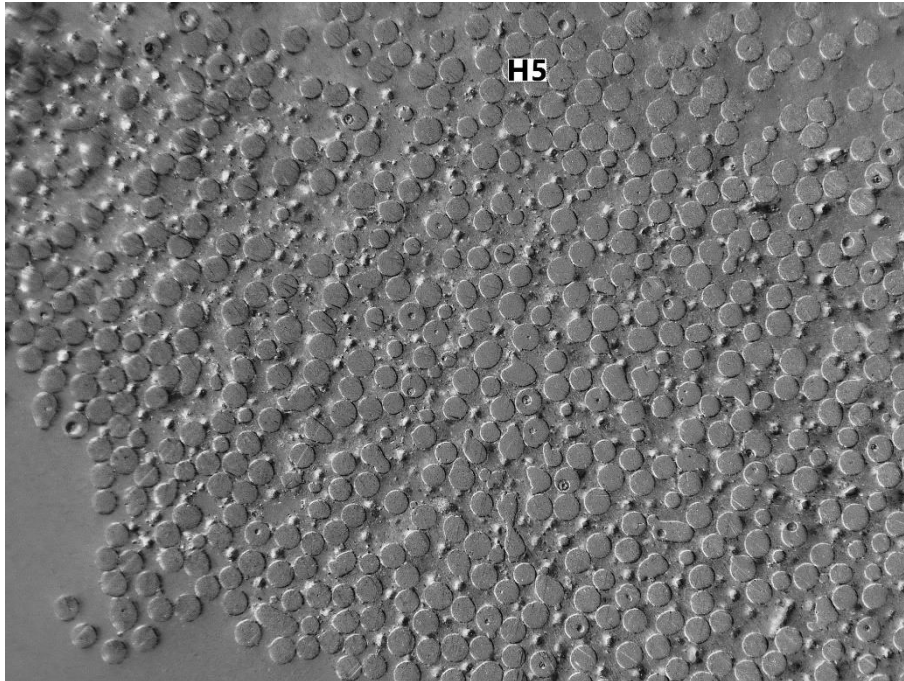


Figure 5:13. Hybrid Polished Particles 106/90 μm (Ref-H5 x100 mag)

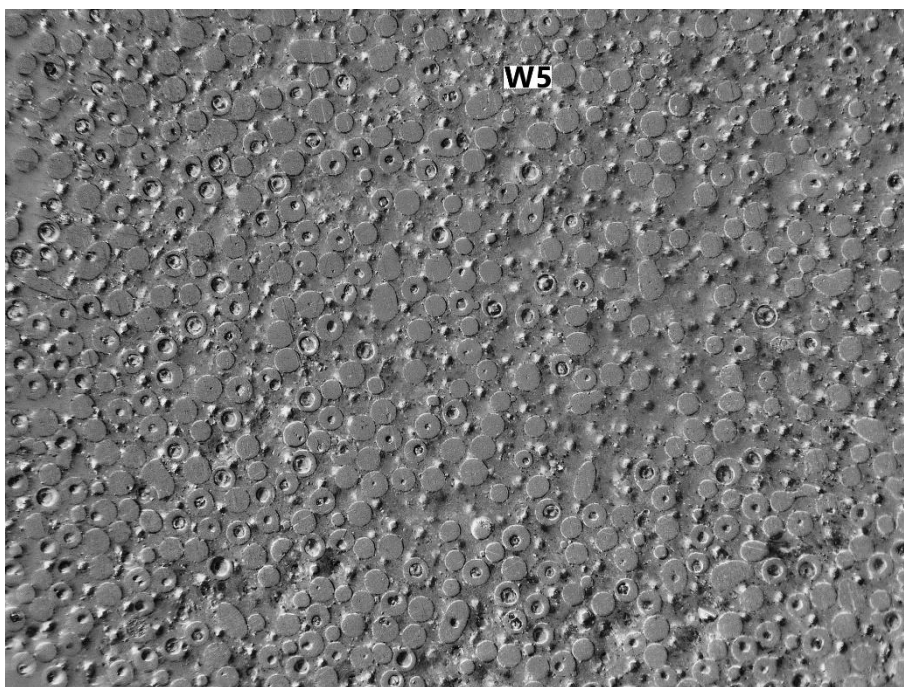


Figure 5:14. Water Polished Particles 106/90 μm (Ref-W5 x100 mag)

5.3.6 Hybrid v Water 90/75 μm (x100 mag)

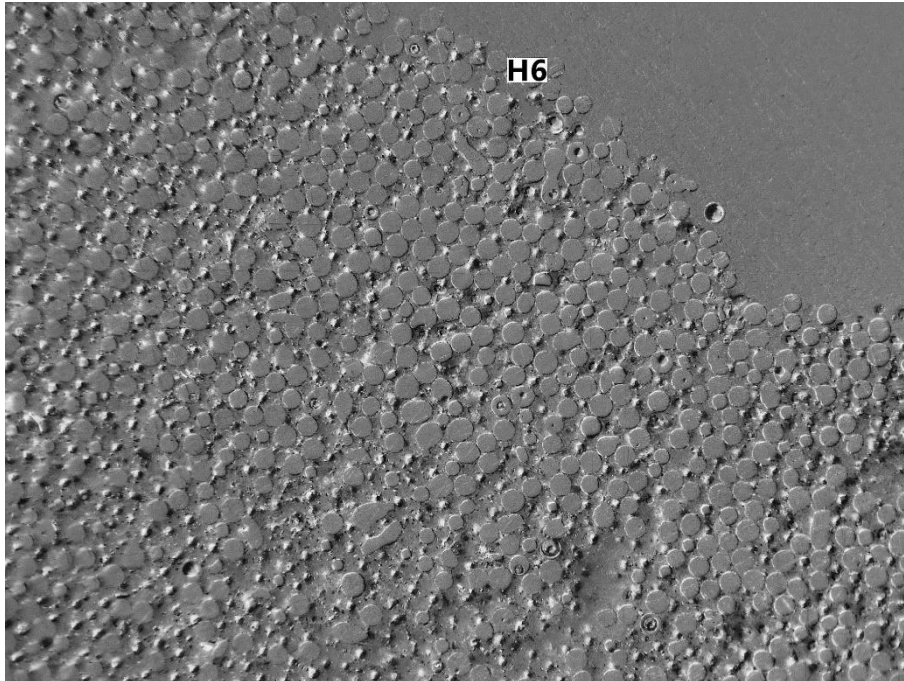


Figure 5:15. Hybrid Polished Particles 90/75 μm (Ref-H6 x100 mag)

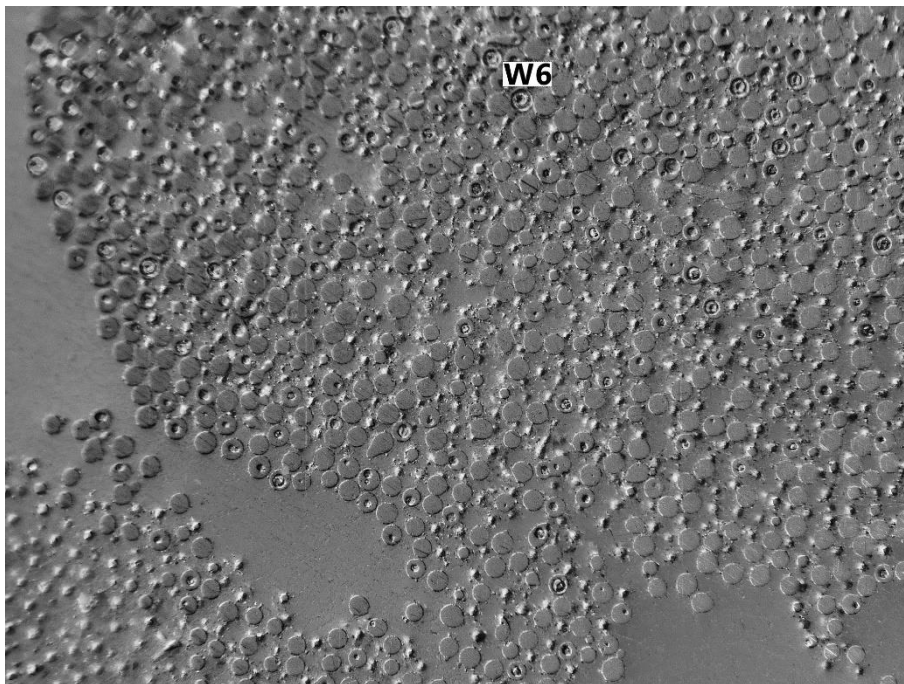


Figure 5:16. Water Polished Particles 90/75 μm (Ref-W6 x100 mag)

5.3.7 Hybrid v Water 75/63 μm (x200 mag)

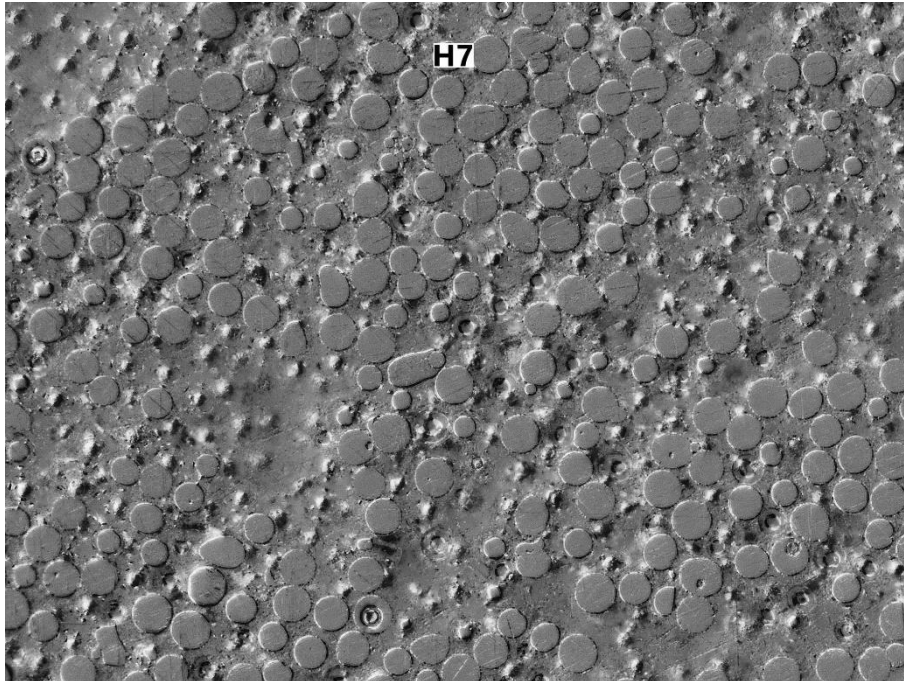


Figure 5:17. Hybrid Polished Particles 75/63 μm (Ref-H7 x200 mag)

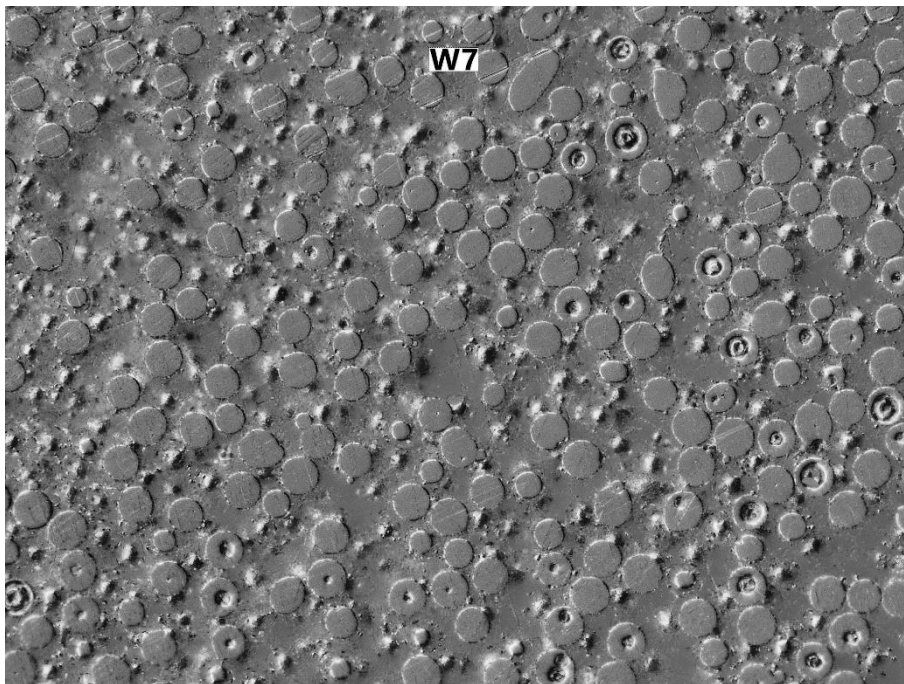


Figure 5:18. Water Polished Particles 75/63 μm (Ref-W7 x200 mag)

5.3.8 Hybrid v Water 63/53 μm (x200 mag)

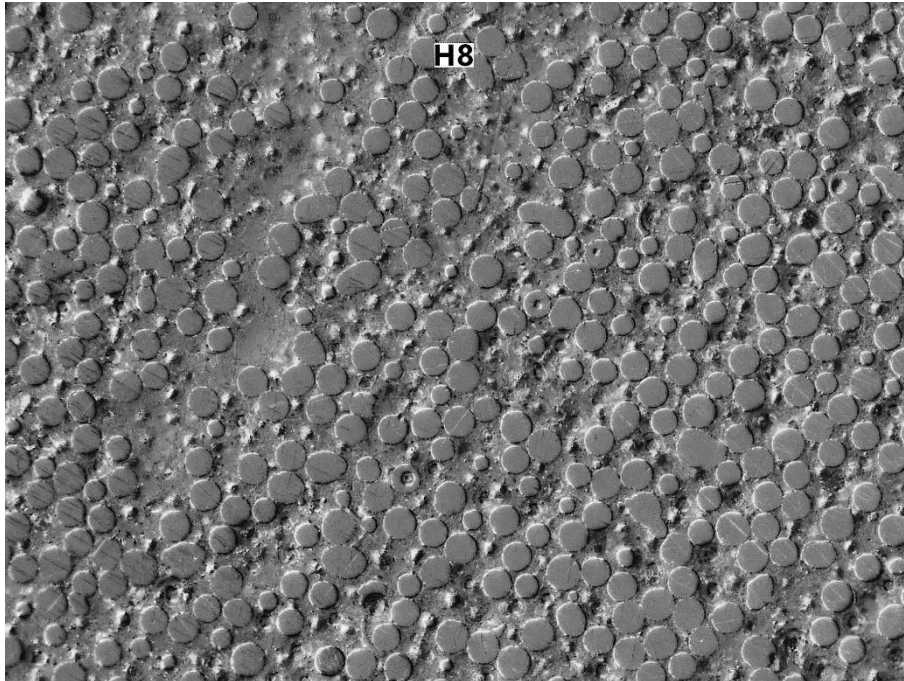


Figure 5:19. Hybrid Polished Particles 63/53 μm (Ref-H8 x200 mag)

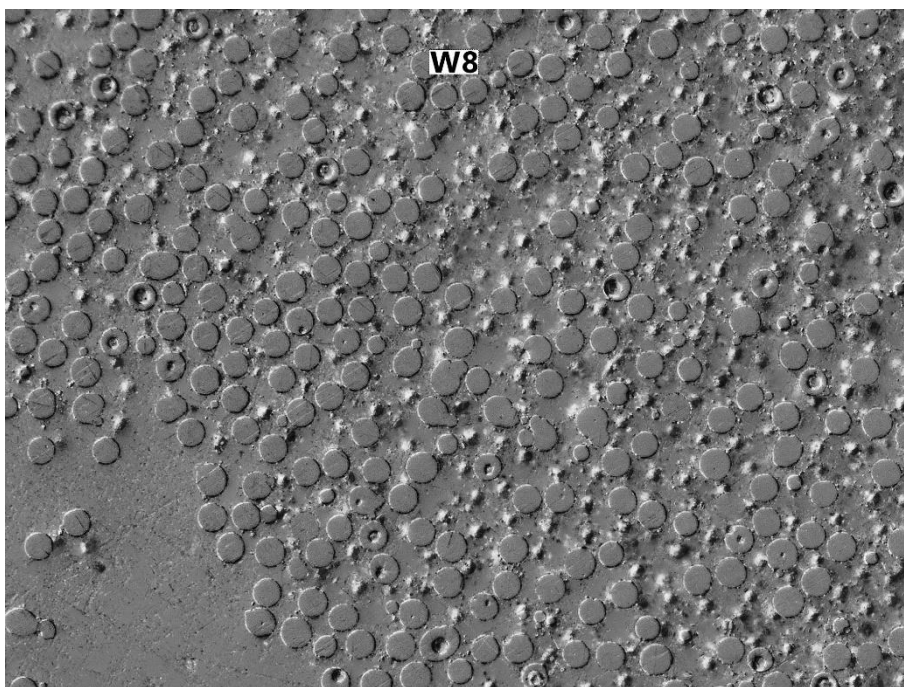


Figure 5:20. Water Polished Particles 63/53 μm (Ref-W8 x200 mag)

5.3.9 Hybrid v Water 53/45 μm (x200 mag)

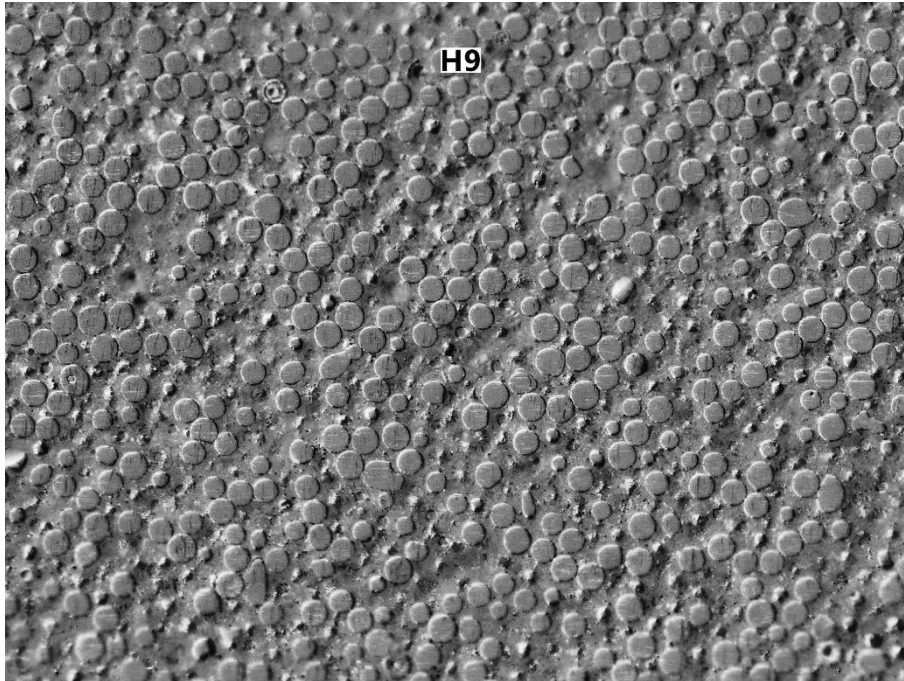


Figure 5:21. Hybrid Polished Particles 53/45 μm (Ref-H9 x200 mag)

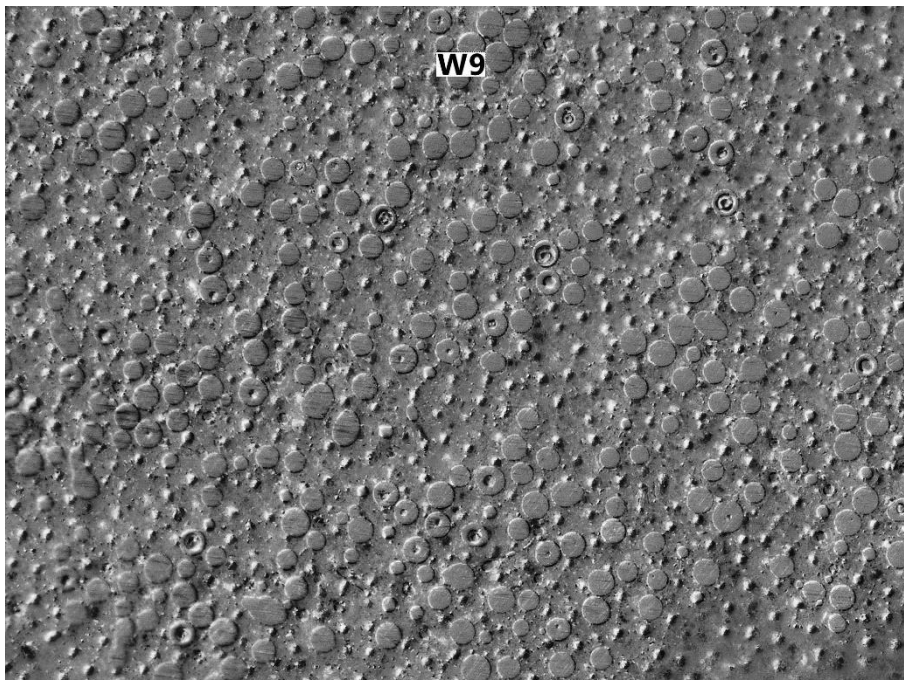


Figure 5:22. Water Polished Particles 53/45 μm (Ref-W9 x200 mag)

5.3.10 Hybrid v Water 45/38 μm (x200 mag)

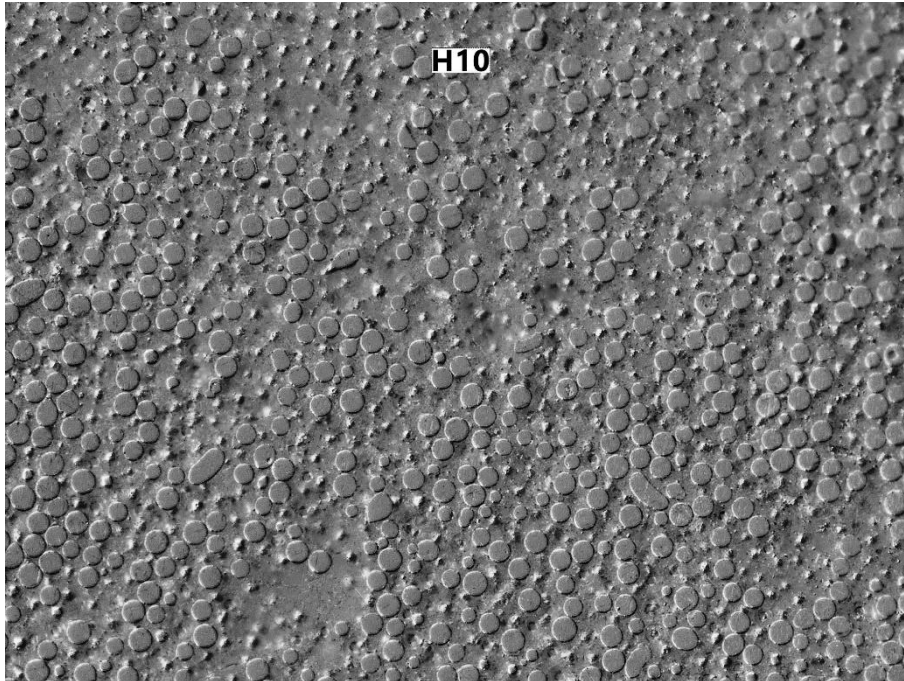


Figure 5:23. Hybrid Polished Particles 45/38 μm (Ref-H10 x200 mag)

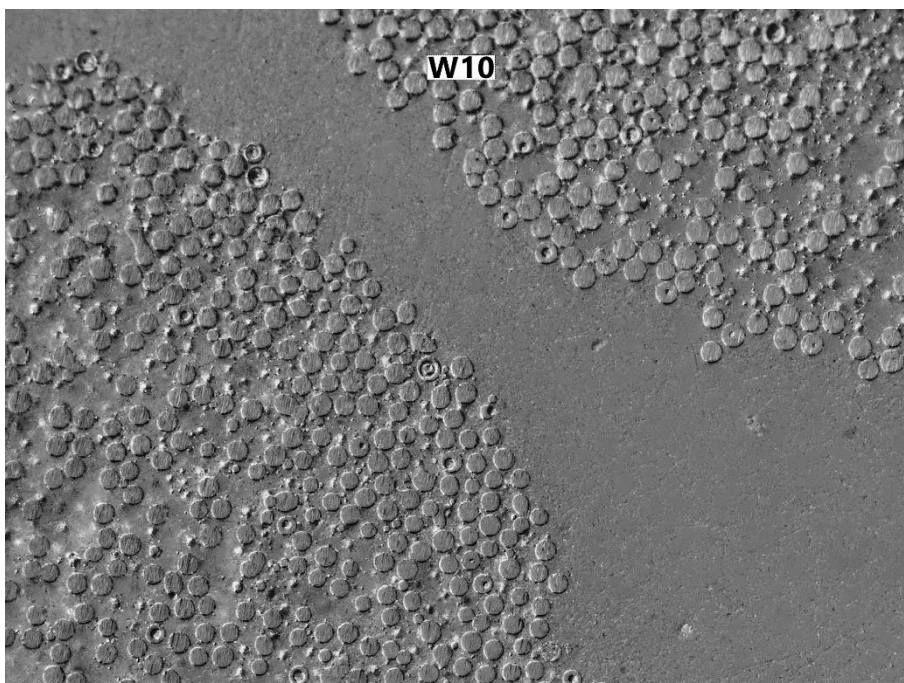


Figure 5:24. Water Polished Particles 45/38 μm (Ref-W10 x200 mag)

5.3.11 Hybrid v Water 38/20 μm (x200 mag)

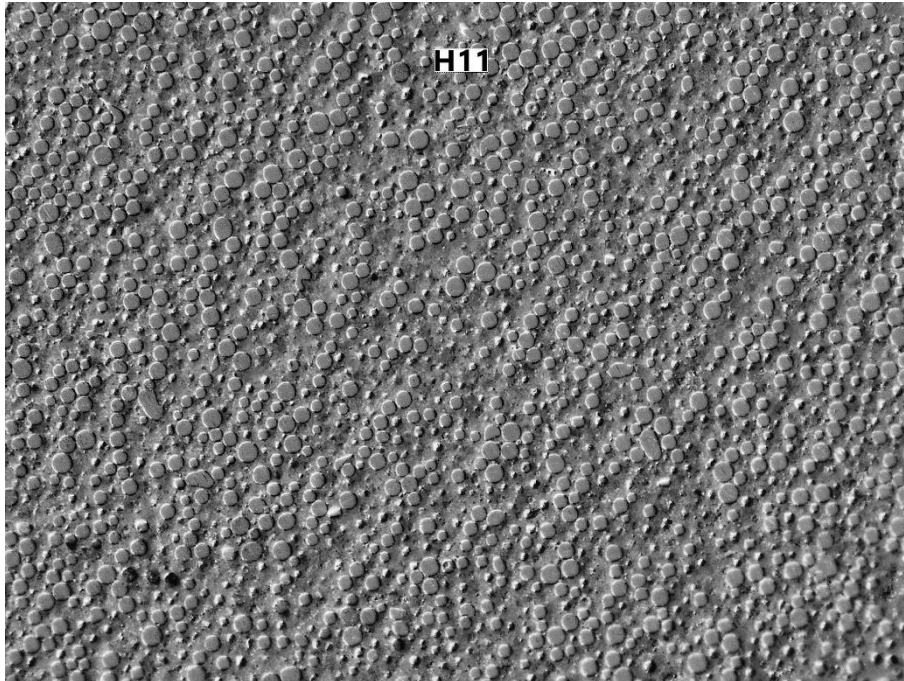


Figure 5:25. Hybrid Polished Particles 38/20 μm (Ref-H11 x200 mag)

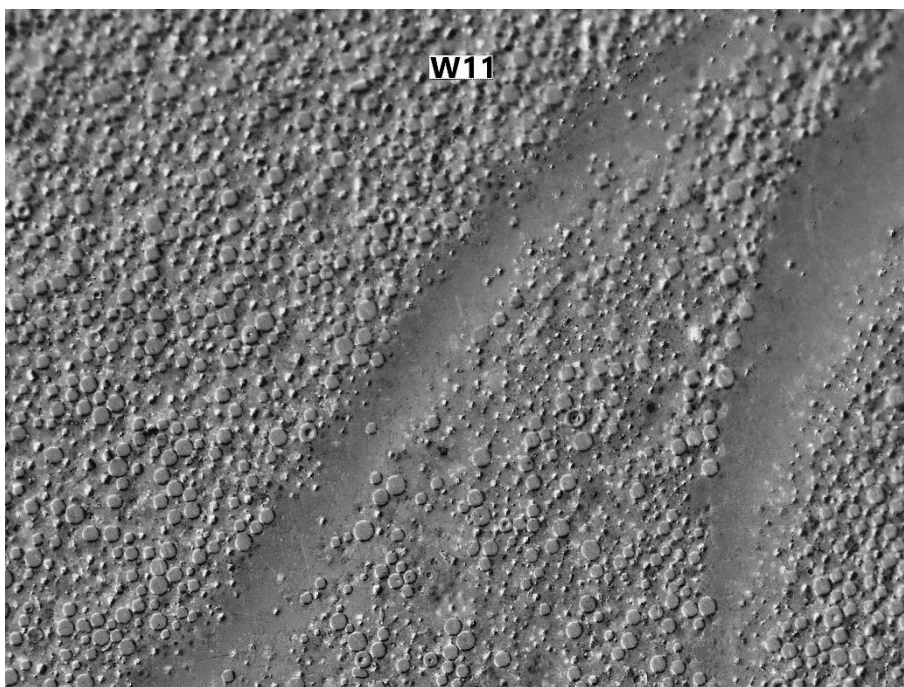


Figure 5:26. Water Polished Particles 38/20 μm (Ref-W11 x200 mag)

5.3.12 Hybrid v Water <20 μm (x400 mag)

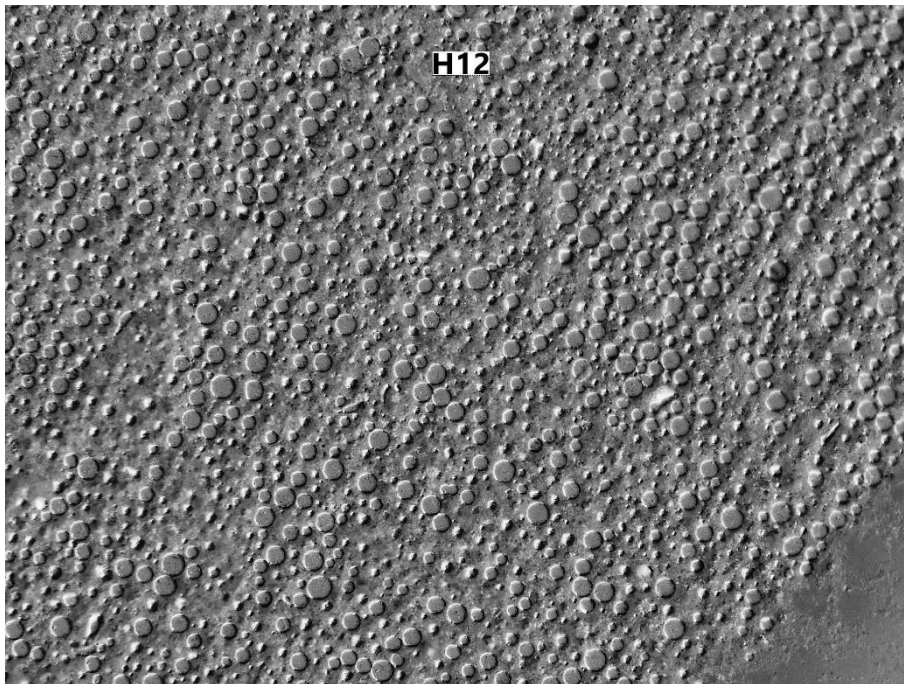


Figure 5:27. Hybrid Polished Particles <20 μm (Ref-H12 x400 mag)

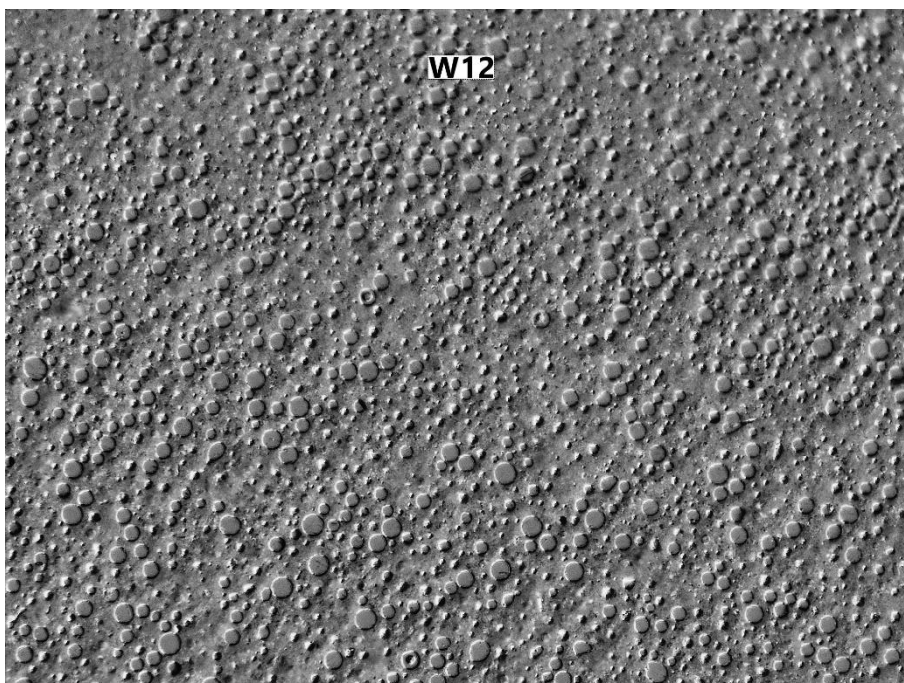


Figure 5:28. Water Polished Particles <20 μm (Ref-W12 x400 mag)

6 Hollow Particle Findings Evaluation of Both Atomization Methodologies

Sample of both hybrid and water atomization material were evaluated (percentage hollow count) and graphs plotted for discussion and conclusion.

6.1 Hollow Particle Findings for Hybrid Atomization

The images taken from the individual /discrete particle size distributions sections clearly shows that less hollow particles (a lower percentage) are present as the hybrid atomized particles become finer in size. A physical hollow count was conducted of each section and results were plotted into a table for evaluation. As per Table 6-1, the percentage values of hollows were calculated by the amount of hollow particles divided by the number of particles which, was photographed on the polished face of the mounted test piece. This particular count was also repeated on another area of the same specimen in an attempt to validate percentage of hollows further. An average of both counts was then used to quantify to final values. From results obtained, the data suggests that as the particles become finer in diameter size, so does the quantity of hollows (trapped gas). The particular section whereby, the final product is between 20/53µm approximately 1.4% of hollow particles are present.

Table 6-1. Hybrid Atomized Hollow Count % by PSD Fraction

Alloy:	N-586X4-LC53			
Lot Number:	Lot:-79256			
Atomising Medium:	Hybrid Atomized:			
Hollow Count:	~ % of Hollow Particles:			
Micron Size Fraction:	Ref No:-	Count 1:	Count 2:	% Average:
>180µm	Ref H1	10	12	11
150-180µm	Ref H2	13	14	14
125-150µm	Ref H3	8	9	9
106-125µm	Ref H4	6	9	8
90-106µm	Ref H5	8	6	7
75-90µm	Ref H6	7	6	7
63-75µm	Ref H7	5	7	6
53-63µm	Ref H8	4	4	4
45-53µm	Ref H9	2	2	2
38-45µm	Ref H10	1	1	1
20-38µ	Ref H11	1	1	1
<20µm	Ref H12	0	1	1

* Equal quantity of particles counted for both count 1 and 2 for average % *

The data shown in Table 6-2 show the actual hollow count of the hybrid atomized material combined sample between 20µm and 53µm whereby the final product is screened/sieved at. A total number of 1936 particles were counted between this particle size range. Summary of finding showed that ~1.4% of hollows particles were present within the specimen.

Table 6-2. Hybrid Summary Hollow Count 20/53µm Test Count 1 & 2 Average

Summary	Hybrid Atomized: 20µm to 53µm Fraction			
Micron Size Fraction:	Ref H9-H11	Count 1:	Count 2:	Total Test 1 + 2
20/53µm (Combined)	Particles Counted	968	968	1936
	Qty of Hollows	13	14	27
	% of Hollows	1%	1%	1.4%

* Equal quantity of particles counted for both test 1 and test 2 for average % *

6.2 Hollow Particle Findings for Water Atomization

The same methodology used in 6.1 in regards, to the hollow count was conducted on the water atomized material. Similar to the hybrid material, as the material becomes finer, less hollow particles are observed. The count of hollow particles is shown in Table 6-3. Although, in this instance with the water atomized material, approximately 3.9% of hollow particles are present between 20/53µ which, is a higher count than the hybrid material. However, for this material to function with little or no detriment to application it is estimated that the material should have no more than approximately 5% of hollow spheres. It is important to note that this suggested 5% max allowance is an assumed industry standard. Again, this is dependent upon the customers particular application.

Table 6-3. Water Atomized Hollow Count % by PSD Fraction

Alloy:	N-586X4-LC53			
Lot Number:	Lot:-79168			
Atomising Medium:	Water Atomized:			
Hollow Count:	~ % of Hollow Particles:			
Micron Size Fraction:	Ref No-:	Test 1:	Test 2:	% Average:
>180µm	Ref W1	70	72	71
150-180µm	Ref W2	61	52	57
125-150µm	Ref W3	57	49	53
106-125µm	Ref W4	36	45	41
90-106µm	Ref W5	34	39	37
75-90µm	Ref W6	23	28	26
63-75µm	Ref W7	15	14	15
53-63µm	Ref W8	13	12	13
45-53µm	Ref W9	8	8	8
38-45µm	Ref W10	3	2	3
20-38µ	Ref W11	2	3	3
<20µm	Ref W12	1	2	2

* Equal quantity of particles counted for both test 1 and test 2 for average % *

Table 6-4 shows the hollow count of ~3.9% between 20µm and 53µm (product area) in the water atomized material. A total number of 1956 particles were counted in this particular instance.

Table 6-4. Water Summary Hollow Count 20/53µm Test Count 1 & 2 Average

Summary	Water Atomized: 20µm to 53µm Fraction			
Micron Size Fraction:	Ref W9-W11	Test 1:	Test 2:	Total Test 1 + 2
20/53µm (Combined)	Particles Counted	978	978	1956
	Qty of Hollows	38	39	77
	% of Hollows	4%	4%	3.9%

* Equal quantity of particles counted for both test 1 and test 2 for average % *

6.3 Hollow Particle Findings Comparison of Both Methodologies

Both sets of data (hybrid and water hollow count) are plotted in Figure 6:1 . From this graph comparison it is clearly evident that as the particles become $\sim >53\mu\text{m}$, the difference in hollow particles between the hybrid and water atomized material increases significantly. Below $\sim 53\mu\text{m}$ and down to $\sim 20\mu\text{m}$ the numbers become similar in percentage of hollows. Finer than $20\mu\text{m}$ (non-product size), majority of the particles both hybrid and water atomized are virtually solid/fully dense with very few hollows. This graph also denotes why other material which is applied by “spray weld” application ($45/125\mu\text{m}$) will be mainly produced by hybrid methodology, as the mean (d50) percentile of water atomized particles contain $\sim 30\%$ hollows which, for this particular spray-weld application, would be detrimental to application. These “thinly” walled particles can burn-up and have the potential to cause porosity in the final applied coating. The consequence of have a porosity or voids in thermal spray coatings include reduction in its mechanical properties and also its susceptibility to corrosion and wear (Ang, A.S.M. & Berndt, C.C., 2014).

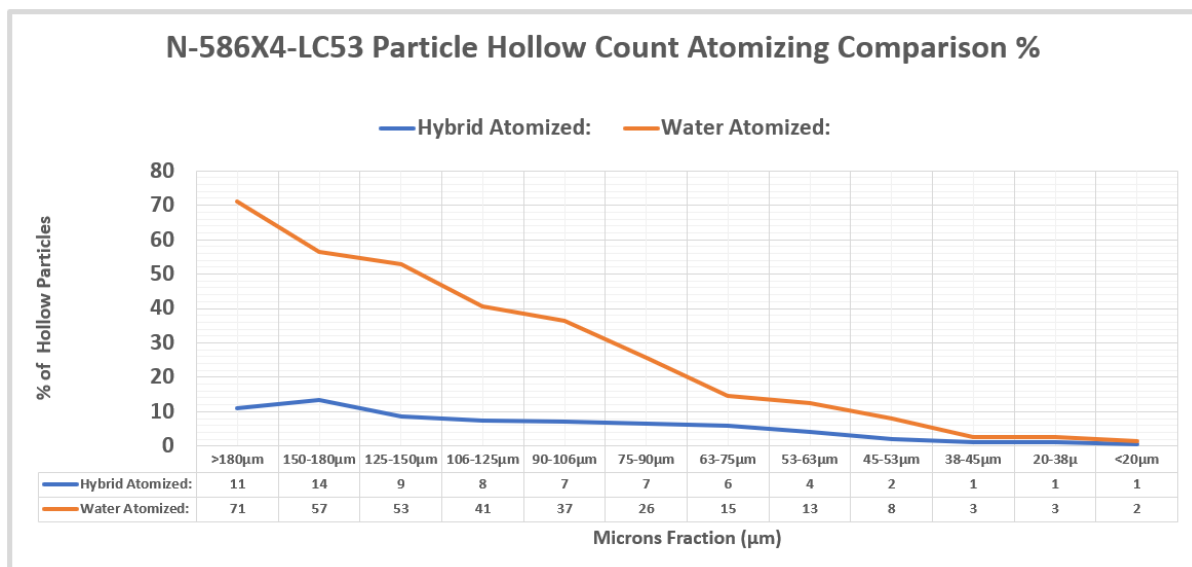


Figure 6:1. Graph % of Hollows per Particle Size Fraction

6.4 Hollow Particle Findings Comparison 20/53 μ m Combined PSD Range

The images shown in Figure 6:2 and Figure 6:3 are the three individual fractions (53/45 μ m) + (45/38 μ m) + (38/20 μ m) physically blended into a combined (53/20 μ m) fraction for both hybrid (lot: 79256) and water atomized (lot: 79168). The percentage summary of hollows counted/quantified are as shown in Table 6-5. The values of water atomized material compare favourably with current production mass produced batches. As mentioned previously, an assumed maximum of 5% have been assumed in the field for producing and acceptable thermal spray coating especially applied by plasma spraying.

Table 6-5. Conclusion of Hollow Spheres by Atomizing Methodology

Av % Hollow Particles Per Product Size Range by Atomizing Medium		
Atomising Medium:	Hybrid Atomized	Water Atomized
Particle Range = 20/53μm	1.4%	3.9%

Fresh samples mounted in resin with sections 20/53 μ m combined for both hybrid and water atomized material were freshly polished and examined. Number of hollows after two individual counts which have been summarised in Table 6-5, virtually match values reported when theoretically combined. Image of these sample can be seen in Figure 6:2 and Figure 6:3

- Hybrid Atomized Powder Freshly Mounted Sample (Particles 20/53 μ m)

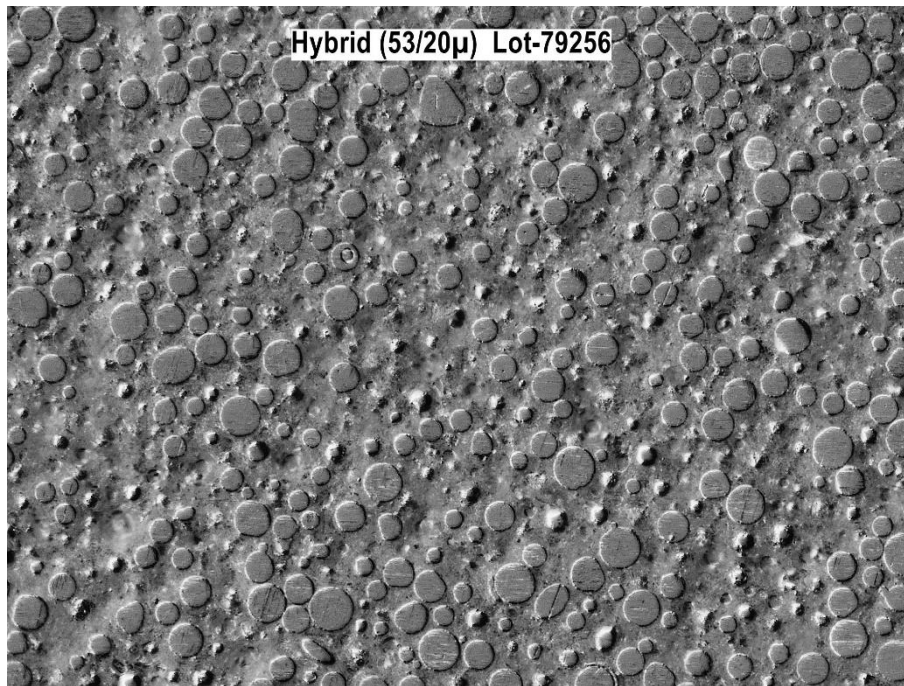


Figure 6:2. Hybrid Particles 53/20 μ m (x300 mag)

- Water Atomized Powder Freshly Mounted Sample (Particles 20/53 μ m)

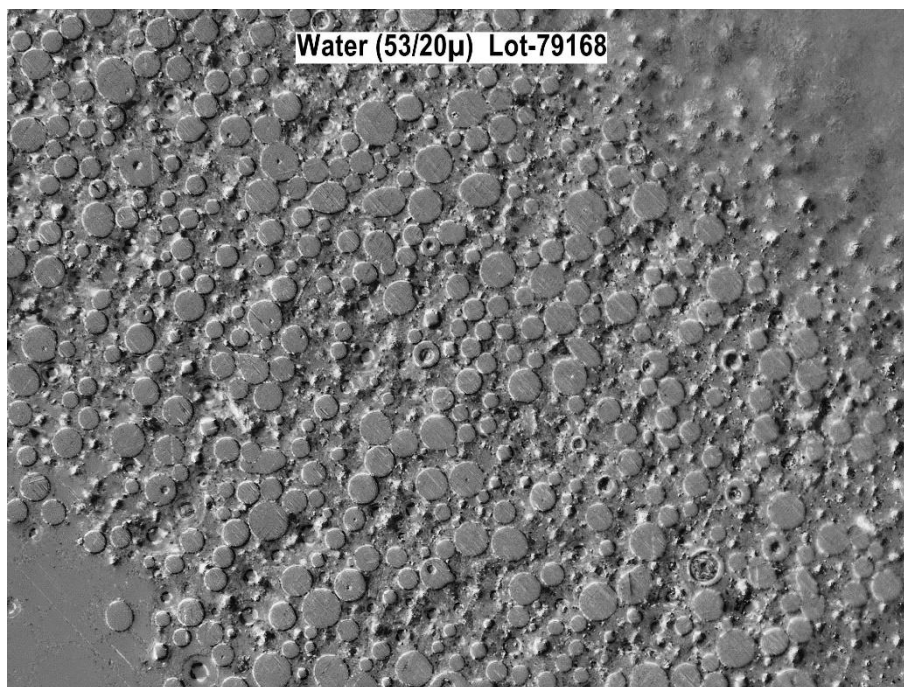


Figure 6:3. Water Particles 53/20 μ m (x300 mag)

6.5 Hollow Count Possible Explanation in Water Atomized Material

Although the hollow count in this instance is acceptable for water methodology atomization due to the theory of “hydrogen” gas formation when atomized with water.

- Theory of Hollows in Water Atomized Powder

Hot molten metal + water = metal oxide + hydrogen.

Chemical Reaction - $M + H_2O = MO_2 + H_2$

According to (*Pasupathy, M. et al, 2016*), fine particles cool/freeze quicker than the larger particles therefore, little time for this chemical reaction and release of H_2 to take place. This theory is mentioned in the book of Dunkley.

Although as mentioned, hollow spheres are inevitable in water atomized material, an attempt to reduce or limit these types of particles by mechanical means were examined. An investigation/study was conducted whereby upon investigation of the water atomized head assembly, the primary jets which, are responsible for atomizing were checked. As shown in Figure 6:5, the jet showed signs of excessive water erosion from the high-pressure water when compared to that of a new water jet. For optimum water atomization, the water jets (both primary and secondary) must centre on the molten metal stream. Any misalignment of jets may cause the molten stream not to be centre thus producing larger droplets which freeze and spheridises as larger “shell-like” particle which, can be classified as “hollow shot” therefore, emphasis of this particular study is to ensure that the water atomization configuration assembly is fully optimized, and the water jets (which, are consumables) are fully operational.

6.6 Water Atomization Jet Set-up Configuration Investigation

Optimization in regards, to the head set-up assembly was discussed in great detail and an Ishikawa fault finding diagram was used as a tool in order, to fully identify areas of process improvements.

6.7 Ishikawa Diagram used for Water Head Set-up Assembly

To optimize the water head set-up, an Ishikawa fish-bone diagram as shown in Figure 6:4, was used at LSN Diffusion in an attempt to identify any particular defects or failures of this dynamic and crucial atomizing configuration set-up. According to *Radziwill, N., (2017)*, an Ishikawa diagram is an analysis tool which, can be implemented and used to explore potential causes in particular defects in industry. This tool also known as a “fishbone” diagram was originated and established back in the early 1960’s by a professor namely, Kaoro Ishikawa whose concept consisted of basic steps that can help to identify the root-causes and help to generate ideas or broader thinking of the issue (*Coccia, M., 2018*). The Ishikawa was used in this instance in an attempt to identify several possible causes rather than using the five whys methodology which, is adept for drilling down to a possible single issue.

Steps usually consist of the following:

- Identification of the issue/problem or original problem.
- Help to identify possible root-cause.
- Identify the particular factors involved in the issue/problem.

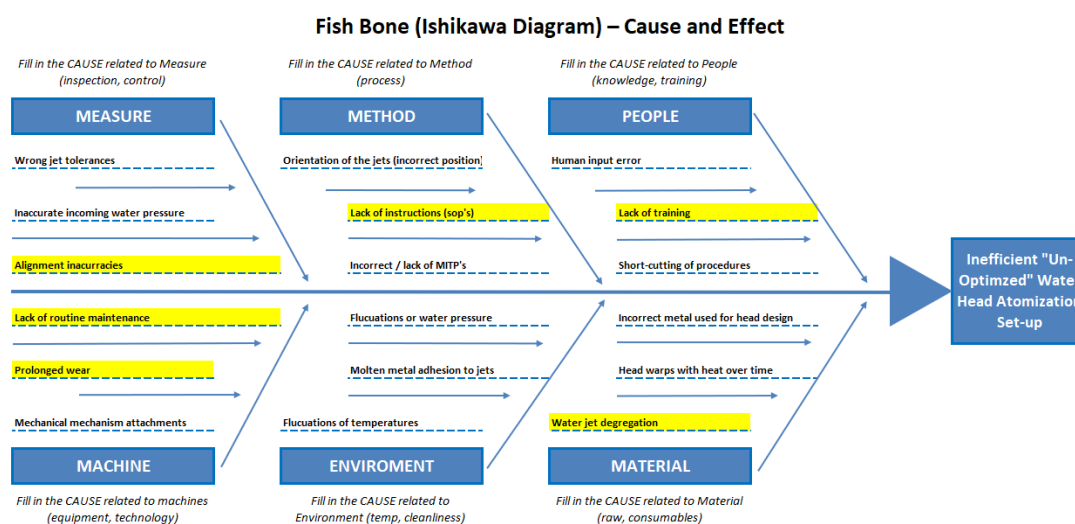


Figure 6:4. Ishikawa "Cause & effect" Used for a Water Head Set-up Assembly

After steps have been taken using the Ishikawa, questions can be asked to “why does potential issue exist or happen”? The diagram in this case highlighted the need to focus on the set-up configuration of the nozzle water jets and the standard operating procedure/training for doing so.

Although there is a water-head drawing detailing the angles/distances of both primary and secondary water jets, there is no specific standard operating procedure (SOP) for operators to follow in regards, to how the set-up should actually be. These water heads assemblies are assembled crudely with little or no accurate precision. Upon investigation of the water primary jets, it appeared that they were extremely eroded by the pressure of the water itself. See Figure 6:5 and Figure 6:6 of a used water jet compared to that of a new water jet Figure 6:7 and Figure 6:8. Water jets are not routinely inspected. However, the water head set-up assembly can be checked on occasions when there are unexpected yield fluctuations and excessive oversize (large hollow metallic particles). These larger, flaky particles tend to float on the collection water in the atomizing chamber.

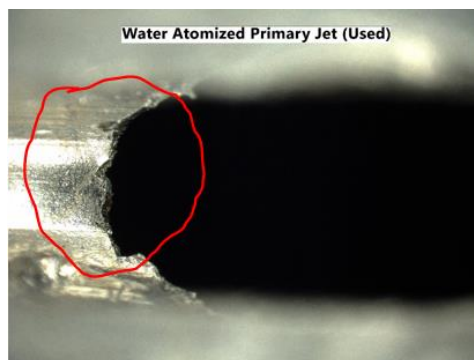


Figure 6:5. "Used" Primary Water Jet (x100 mag)



Figure 6:6. Used Primary Water Jet

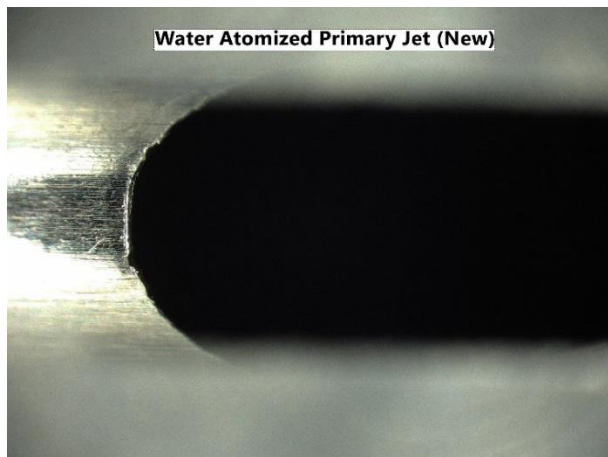


Figure 6:7. "New" Primary Water Jet (x100 mag)



Figure 6:8. New Primary Water Jet

From these images taken by the Keyence microscope it is evident that the water jet/spray would be deflected “away” from the dead-centre of molten metal by the worn/eroded jets. This has the potential to create large powder shot-type low density hollow type particles which, indeed was observed when the water atomised particles $>180\mu\text{m}$ were subjected to the hollow count.

6.8 Hollow Count Using New Water Primary Jets

A fresh batch of material was produced using new/replaced primary atomizing jets. The water head set-up was according to the dimensions of the water assembly collar drawing as per Figure 6:9. A full hollow check per particle size fractions were freshly prepared so a comparison between hollow count can be evaluated between old and new primary water jets.

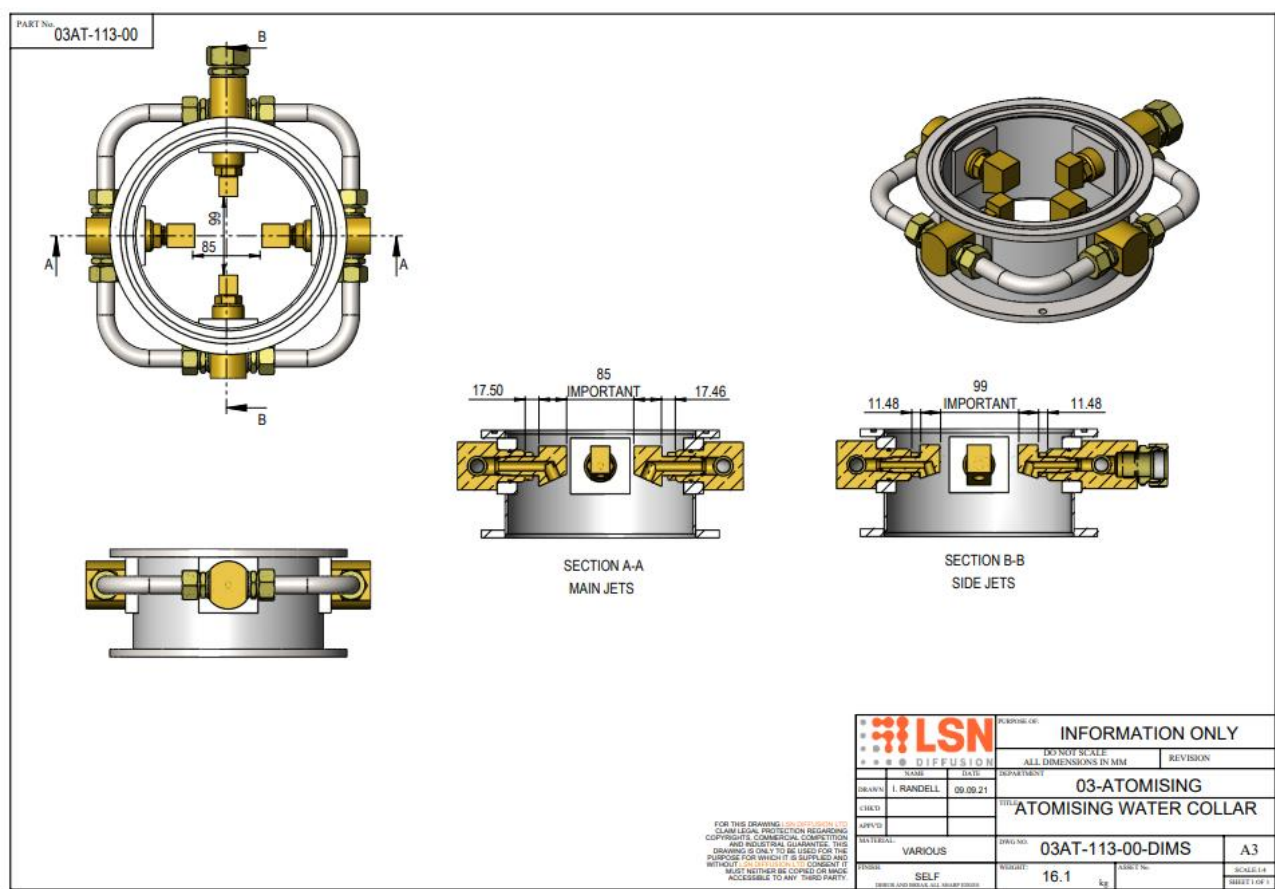


Figure 6:9. Water Assembly Set-up Actual Drawing

As detailed in Table 6-6, a physical particle hollow count was undertaken on each section (similar to previous counts) of the freshly produced material for comparison purposes against the values taken from material which, had defective or water jets. Data collected from material using new water jets show a lower percentage of hollow particles as the particles become finer in diameter size. Again, this count was repeated on the same specimen mounted sample in an attempt to validate further initial count. However, in this case the final product size 20/53µm shows that ~3.1% of hollow particles are present in the sample compared to ~3.9% previously. Therefore, a reduction of hollow type particles of nearly 1% (20% relative) between material of old and used primary atomizing jets.

Table 6-6. Water Atomized Hollow Count Using "NEW" Primary Jets % by PSD Fraction

Alloy:	N-586X4-LC53 (New Jets)			
Lot Number:	Lot:79759 (New Jets)			
Atomising Medium:	Water Atomized: (New Jets)			
Hollow Count:	~ % of Hollow Particles:			
Micron Size Fraction:	Ref No-:	Test 1:	Test 2:	% Average:
>180µm	Ref W1	55	52	54
150-180µm	Ref W2	54	45	50
125-150µm	Ref W3	38	40	39
106-125µm	Ref W4	29	31	30
90-106µm	Ref W5	21	29	25
75-90µm	Ref W6	21	19	20
63-75µm	Ref W7	12	11	12
53-63µm	Ref W8	7	9	8
45-53µm	Ref W9	6	5	6
38-45µm	Ref W10	3	2	3
20-38µ	Ref W11	1	2	2
<20µm	Ref W12	2	1	2

* Equal quantity of particles counted for both test 1 and test 2 for average % *

Summary	Water Atomized: 20µm to 53µm Fraction			
Micron Size Fraction:	Ref W9-W11	Test 1:	Test 2:	Total Test 1 + 2
20/53µm (Combined)	Particles Counted	1072	1072	2144
	Qty of Hollows	31	35	66
	% of Hollows	3%	3%	3.1%

* Equal quantity of particles counted for both test 1 and test 2 for average % *

Comparison between water atomization methodologies (old v new jets) as shown in Table 6-7, clearly shows a reduction of nearly 1% (~20% overall reduction relatively) between the product final particle distribution of 20/53 μ m which, is quite significant.

Table 6-7. Comparison of % Hollows of Water Atomized Product (Original set-up v New Jets)

Av % Hollow Particles Comparison of Both Water Atomized 79168 & 79759		
Atomising Medium:	Water Atomized	Water Atomized (New Jets)
Particle Range = 20/53 μ m	3.9%	3.1%

Values obtained from the individual hollow count using new jets were compared with initial counts with that of the used/defective water jets and also of the hybrid atomized material as shown in Figure 6:10. From the profile it is clear that there is an overall reduction of hollows using the refined water atomized material. Although, both sets of water atomized material are still significantly higher than that of the hybrid atomised material especially on the coarser particle sized of the material >53 μ m. However, there is a clear shift of solid/dense particles sub 53 μ m which suggests that the newly produced material has improved in quality.

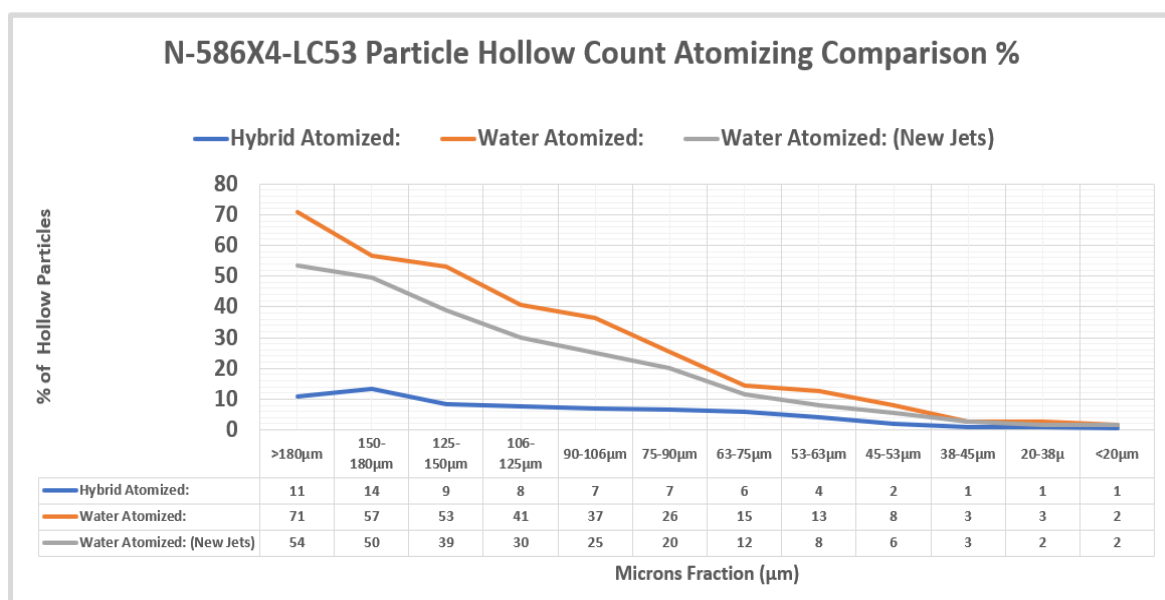


Figure 6:10. Graph % of Hollows per Particle Size Fraction for All Tests

6.9 Water Atomization Primary Jets Investigation Conclusion

It appears that by using the Ishikawa methodology, issues were highlighted regarding the water head set-up. These issues were examined, and it was discovered that the primary water jets were subject to deterioration via the high velocity/pressure of the water. From this research done, a standard operating procedure (SOP) should be completed detailing the set-up of the water collar assembly as well as all the specific instructions of how this preventative/routine maintenance work should be completed. This would also include the replacement of both primary and secondary water jets (consumable parts) periodically subject to further research being done which, would define the timescale of doing so. Although, changing the primary water jets did not resolve the issue of hollow particles it did reduce the overall percentage by approximately 20% relative.

A screen analysis on both material samples clearly shows a shift to the finer side with the material using new primary jets. This material has a lower mean size. However, the theoretical screening yield between both powders are not too dissimilar. See Figure 6:11. It appears that by changing the primary water jets, less deflection away from molten stream therefore, the water spray is more effective by not producing so much coarse particles which, as demonstrated produces the most hollows. Although, the reduction of coarser particles was off-set due to the material containing additional fine material (Sub 20 μ) thus theoretical yield remaining similar.

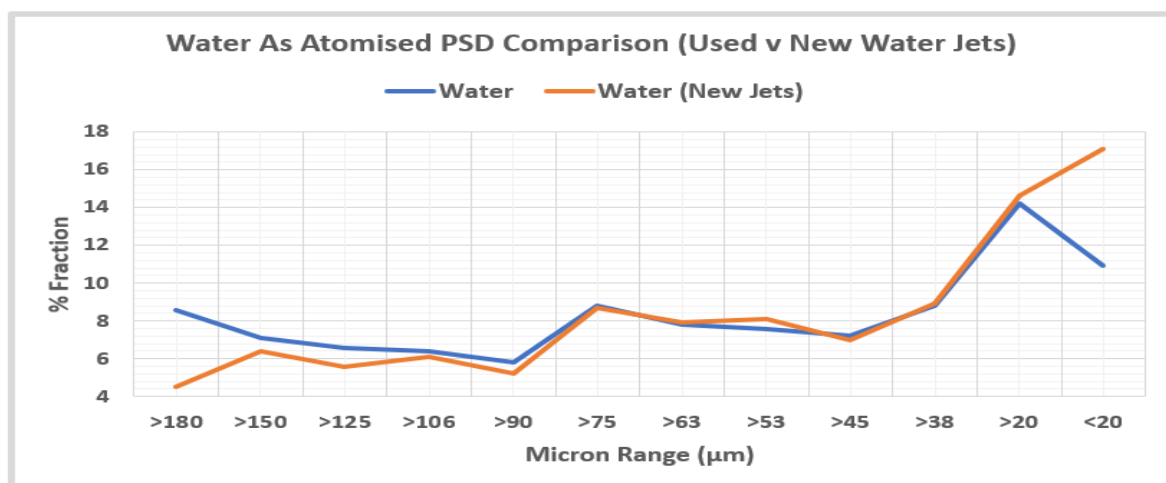


Figure 6:11. Particle Size Distribution Plots of both Water Lot-79168 & 79759 (as atomized)

6.10 Hollow Count Conclusion of Both Methodologies

All data gathered from both hybrid and water atomizing methodologies show that the hybrid material contain fewer hollow particles than that of the water atomized material, even after changing the primary water jets. Summary of this data can be seen in Table 6-8. More research and development should be undertaken going forward. This may include, varying the water head set-up configurations such as, water jet angles, nozzle protrusion, nozzle diameter and water pressure. By re-configuring these parameters, multiple permutations may be possible for reducing the hollow count further. This may, however, have an impact upon yield.

Table 6-8. Summary Hollow % Count of Atomizing Methodologies

Atomizing Methodology:	Hollow Count %:
Hybrid Atomized	1.4%
Water Atomised	3.9%
Water Atomised (optimum Jet Set-up)	3.1%

The water atomization parameters for this particular alloy as per Table 6-9 have the following parameters. The water jets referenced 25/8, predominately produce fine powder which, are typically used for other high yielding hard-surfacing material ranging from 106/20µm.

Table 6-9. Current Water Atomization Parameters

Jet Configuration:	Nozzle Diameter:	Water Pressure (psi) at Head
25 / 8's (suitable for fine powder)	6.0 mm	120 bar

7 Yield Considerations for converting from Hybrid to Water for N-586

As atomized samples (un-screened material) were taken from both of the hybrid and water batches used in this study and subjected to a screen analysis test. From the data as shown in Table 7-1, and also as shown in Figure 7:1 and Figure 7:2. It is clear that there is substantially more product with the water atomized material between 53 & 20 μ m. Data shown in Table 7-2 and Figure 7:3 also supports this as many screen tests were conducted on water atomizing methodologies over a period of time. Values reported imply that the water atomization method is superior in regards, to a higher theoretical yield than that of the as atomized hybrid material.

As discussed, the hybrid atomized material using the fine insert produces the most product with the customer requested particle size distribution between 53 & 20 μ m. However, other various gas head configurations can be used such as the free-low dual stage head. These configurations, however, are tailored to produce more material between the bands 53 and 125 μ m which in turn, has a higher yield than that over the water atomised material. It is important to note however, that although the water atomized material is cheaper to produce, when producing spray weld or plasma transferred arc (PTA) type material for example, the hybrid route would be preferred due to its higher yield between these specific particle size bands that off-sets the costs and also most importantly, the higher percentage of solid particles which, are found with this atomizing methodology. Having excess hollow particles in a PTA type material can be detrimental to application due to the lower melting temperature which these particles melt. Excessive hollow spheres when applied can cause porosity within the coating when applied to the base-metal (substrate).

7.1 As Atomized Yield Comparison Data

As shown in Table 7-1, the theoretical and screening yield is significantly higher with the water atomized material for this specific customer application. This was also the case when new water jets were used to potentially reduce the number of hollow particles. Data suggests that water atomization product has an approximate 30% relative increase in theoretical yield. The screening efficiency yield (final yield after sieving) also has a similar relative increase in yield.

Table 7-1. "As Atomised" Particle Size Distribution of both lots: Hybrid 79256 & Water 79168.

	As Atomised Screen Analysis Methodology Comparison	
Alloy:	N-586X4-LC53	N-586X4-LC53
Lot Number:	79256	79168
Atomizing Medium:	Hybrid	Water
Atomizing Assembly:	Fine Insert (Tapered)	Square 25 / 8's
Atomizing Nozzle:	6 mm	6 mm
Atomizing Pressure:	300 Psi	120 bar
>180	6.8	8.6
>150	6.4	7.1
>125	10.2	6.6
>106	10.1	6.4
>90	9.8	5.8
>75	7.2	8.8
>63	9	7.8
>53	7.4	7.6
>45	6.5	7.2
>38	6.8	8.8
>20	7.8	14.2
<20	11.7	10.9
Total	99.7	99.8
~Theoretical Yield (53/20µm)	21.1	30.2
Less ~20% Inefficient Screening	~17%	~24%

Figure 7:1 represents the theoretical product yield difference between the hybrid and water atomized material. This graph clearly shows in greater detail, the product area for hybrid (20/38 μ), is inferior in regards, to the quantity of saleable product than that of the water produced material.

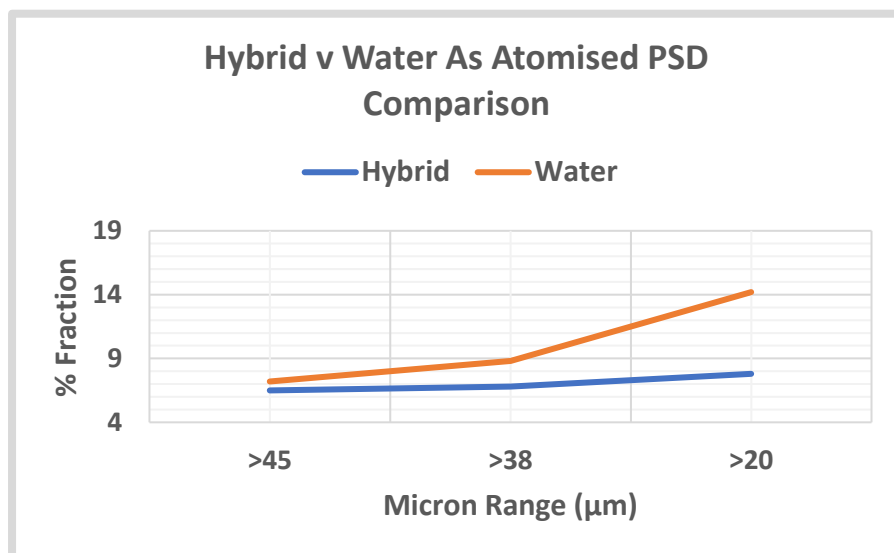


Figure 7:1. Difference in % Between Hybrid & Water (53/20 μ)

These values as seen in Figure 7:2 is a graphical and visual representation of the values obtained in the overall particle distribution in regards, to the water methodology which, also shows a higher proportion of product between 53 & 20 μm .

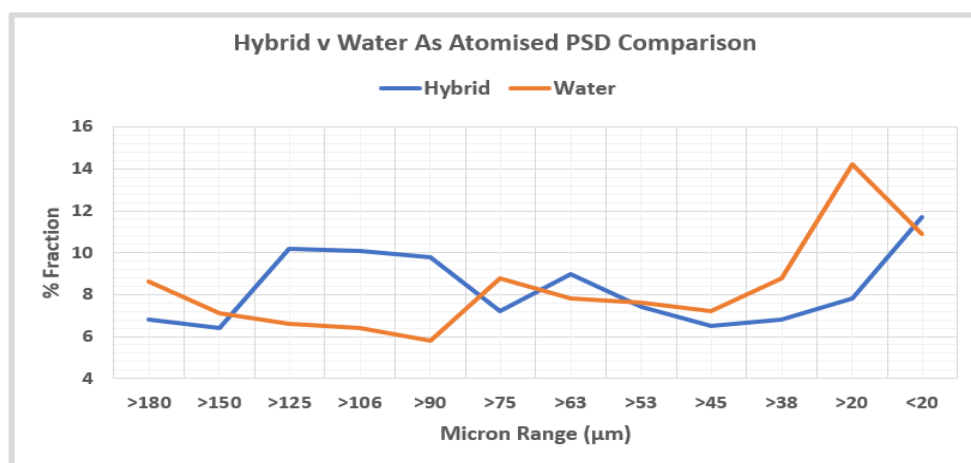


Figure 7:2. Plots of both lots: Hybrid 79256 & Water 79168 (as atomized)

7.2 Consistency of Theoretical Yield of Water Atomized Material

Further batches of water atomized were produced and subjected to as atomized theoretical yield predictions which are logged in Table 7-2. This data shows the consistency yield of the water atomized product.

Table 7-2. Water Atomized "As Atomised" Theoretical Analysis Taken from Several Lots

Alloy:	N-586X4-LC53	N-586X4-LC53	N-586X4-LC53	N-586X4-LC53
Lot Number:	79107	79245	79709	79758
Atomizing Medium:	Water	Water	Water	Water
Atomizing Assembly:	Square 25 / 8's	Square 25 / 8's	Square 25 / 8's	Square 25 / 8's
Atomizing Nozzle:	6 mm	6 mm	6 mm	6 mm
Atomizing Water Pressure:	200 bar	200 bar	200 bar	200 bar
>180	11.1	10.5	8.1	9.5
>150	7.6	8.0	6.8	7.3
>125	6.9	5.9	6.2	6.6
>106	6.5	5.9	6.4	6.5
>90	6.3	7.1	5.5	6.4
>75	6.4	6.9	7.6	7.0
>63	7.6	7.2	7.7	6.9
>53	7.4	6.3	6.8	6.7
>45	6.9	7.0	5.7	7.2
>38	8.6	9.0	7.7	9.4
>20	13.9	11.6	14.5	14.8
<20	10.5	15.0	15.9	11.5
Total	99.7	100.4	98.9	99.8
~Theoretical Yield (53/20 μ m)	29.4	27.6	27.9	31.4
Less ~20% Inefficient Screening	~23%	~22%	~22%	~25%

Figure 7:3 shows the consistency of theoretical screening yield (53/20 μ m) of all of the four random batches of water atomized material which, indicates the current stability of the manufacturing process. This specific data has been extracted from Table 7-2 for a clearer picture of this particle size range.

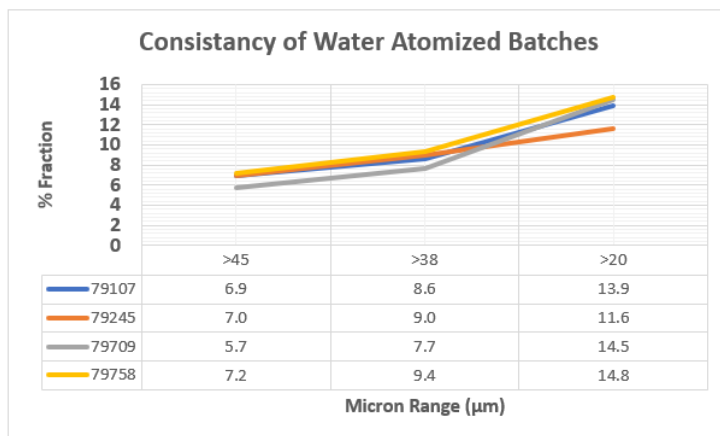


Figure 7:3. Water Atomized Material Consistency (53/20 μ m)

This data also compares favourably with initial reported yield values of the material with the replacement jets which, contains reduced hollow particles. The data in Table 7-3, shows this reported shift of particles towards the finer end (sub 20 μ) but has also less material on the coarse end (>180 μ). Although, the product between 20 μ m and 53 μ m (theoretical product yield area) remains unchanged.

Table 7-3. "As Atomised" PSD Comparison of Water Lots 79168 & 79759 (old v new jets)

As Atomised Screen Analysis Methodology Comparison		
Alloy:	N-586X4-LC53	N-586X4-LC53
Lot Number:	79168	79759
Atomizing Medium:	Water	Water (New Jets)
Atomizing Assembly:	Square 25 / 8's	Square 25 / 8's (New Jets)
Atomizing Nozzle:	6 mm	6 mm (New Jets)
Atomizing Pressure:	200 bar	200 bar (New Jets)
>180	8.6	4.5
>150	7.1	6.4
>125	6.6	5.6
>106	6.4	6.1
>90	5.8	5.2
>75	8.8	8.7
>63	7.8	7.9
>53	7.6	8.1
>45	7.2	7.0
>38	8.8	8.9
>20	14.2	14.6
<20	10.9	17.1
Total	99.8	100.1
~Theoretical Yield (53/20 μ m)	30.2	30.5
Less ~20% Inefficient Screening	~24%	~24%

Figure 7:4 shows in more detail theoretical yield comparison of the product range (53/20 μ) area of the material atomized water using old and new water jets. Both samples show a similar theoretical yield.

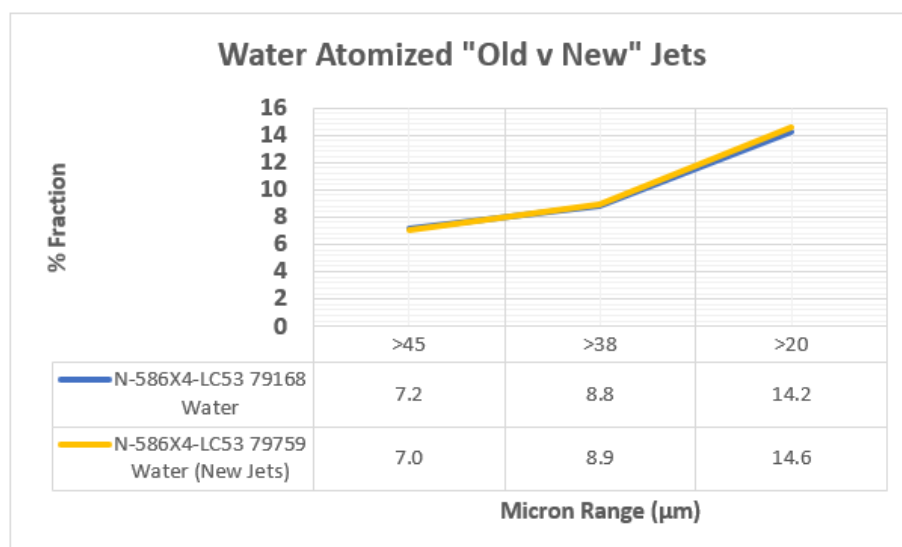


Figure 7:4. Water Atomized Old v New Theoretical Values % (53/20 μ)

7.3 Laboratory Evaluation of Finished Screened Material

The laboratory evaluation data of both products, as per Table 7-4, shows that after processing using low energy rotary sieving equipment with powder classifies both hybrid, and water atomized batches meet the internal specification using the same processing parameters. The actual yields of both products are also reported in the data. Both yields on the finished product are relative to the “as atomized” theoretical reported values. Although, the water atomized yield in fact on this particular lot-79168 is slightly higher than estimated values (after insufficient screening which is taken into consideration). The sub 20µm & sub 16µm values, however, are normally measured using microtrac, laser technology and added to the values obtained by the “physical” sieve measurements which, are normally above 38µm. Laser sizing evaluates the powder particles by shape and by size. If particles are not 100% spherical then the laser sizer would pick these-up as larger in diameter than what the particle is measured when “physically” sieved to size using aperture size screens therefore, a negative correlation between laser and physical sieve measurements. This is the ramification of measuring particles by laser if the particles are not fully spheroidised. The finished screening yields were 18% and 26% respectively. Therefore, clearly the water atomized material has significantly a higher of around ~25-30% relative in yield improvement.

Table 7-4. Final Laboratory Evaluation Hybrid v Water Atomization

Final Material Evaluation				Product Specification
	Alloy:	N-586X4-LC53	N-586X4-LC53	N-586X4-LC53
	Lot Number:	79256	79168	
	Atomizing Medium:	Hybrid	Water	
Chemical Composition:	B	3.02	3.13	2.8 / 3.2
	C	0.68	0.68	0.55 / 0.75
	Cr	15.72	15.56	15.0 / 16.0
	Fe	3.74	3.53	2.4 / 4.6
	Others	0.18	<0.10	< 0.2
	P	0.01	0.01	0.1 max
	Si	4.14	3.99	3.9 / 4.5
	O	0.05	0.06	0.08 max
	N	<0.01	<0.01	0.03 max
Particle Size Distribution:	>75	0.0	0.0	Nil
	>63	0.4	0.2	0.5 Max
	>53	3.1	1.2	1.0 / 5.0
	>45	20.2	17.5	15.0 / 25.0
	>38			
	>20	Balance	Balance	Balance
	<20	2.0	1.6	1.5 / 3.5
	<16	Trace	0.1	0.1 Max
Physical Characteristics:	Hardness HRc	57.3	57.1	56 / 62 Rc
	Apparent Density gm/cm ³	4.4	4.4	> 4.2
	Hall Cup Flow (secs)	14.4	14.8	< 18 secs
Product Yield:	Actual Product Yield %	18%	26%	N/A

8 Product Deposition Comparison of Hybrid V Water Atomised Product

Both hybrid and water atomized material samples of N-586 were deposited on a small mild-steel test piece for a comparison evaluation as shown in Figure 8:1. Sample was then ground down using a swing grinder and polished using a various sized grit paper. This sample specimen was then subjected to further metallography preparation by the use of a diamond compounds on lapping polishing cloths until a mirror-like finish could be observed. Half the deposited test piece of was etched in an acid solution so a more detailed grain structure as suggested by *Radzikowska, J., (2000)*, could be more visibly seen/pronounced.

Using the digital imaging equipment available to us such as the Keyence, an estimation in regards, to the porosity count can then be determined both of the atomizing methodologies.

8.1 Deposit of Hybrid and Water Material



Figure 8:1. Deposition of Hybrid and Water Atomized Material

8.2 Hybrid Atomised Metallography / Grain Structure and Deposition Porosity Count

Two photographic images were taken using the Keyence microscopic equipment of the hybrid deposited material can be seen in Figure 8:2 and Figure 8:3. Whereby areas of which, are circled have been identified of have porosity of some sort.

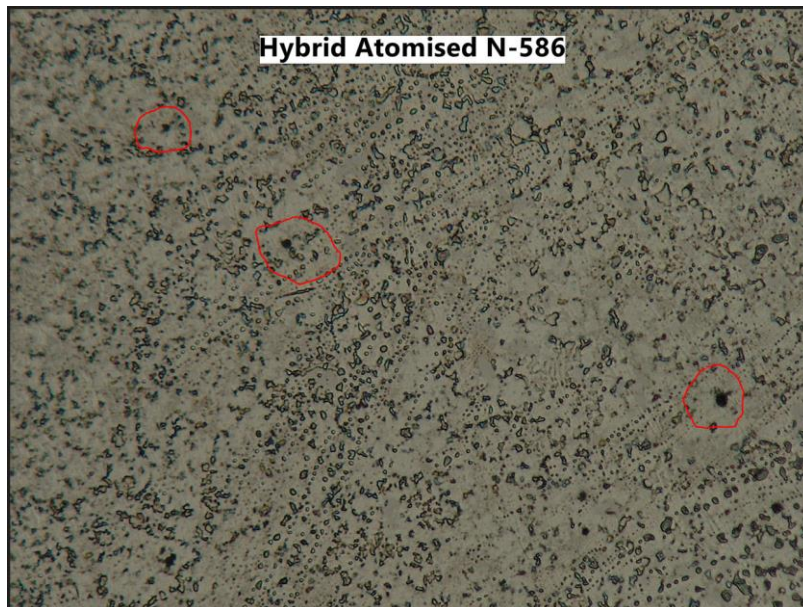


Figure 8:2. Metallography Grain Structure Image Deposition (x1000) Hybrid Material

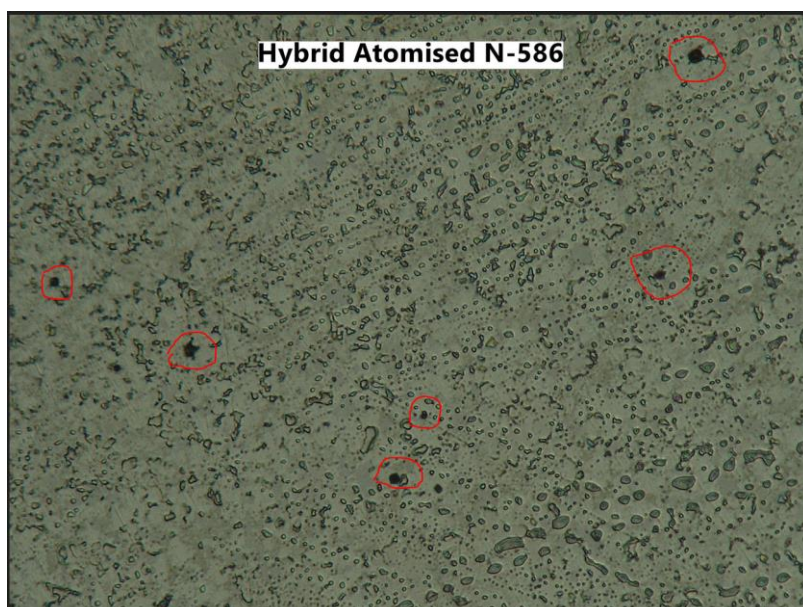


Figure 8:3. Metallography Grain Structure Image Deposition (x1000) Hybrid Material

8.3 Water Atomised Metallography / Grain Structure and Deposition Porosity Count

Two photographic images were taken using the Keyence microscopic equipment of the water atomized deposited material can be seen in Figure 8:4 and Figure 8:5. Whereby, areas which, are circled have been again, identified of have some sort of porosity.

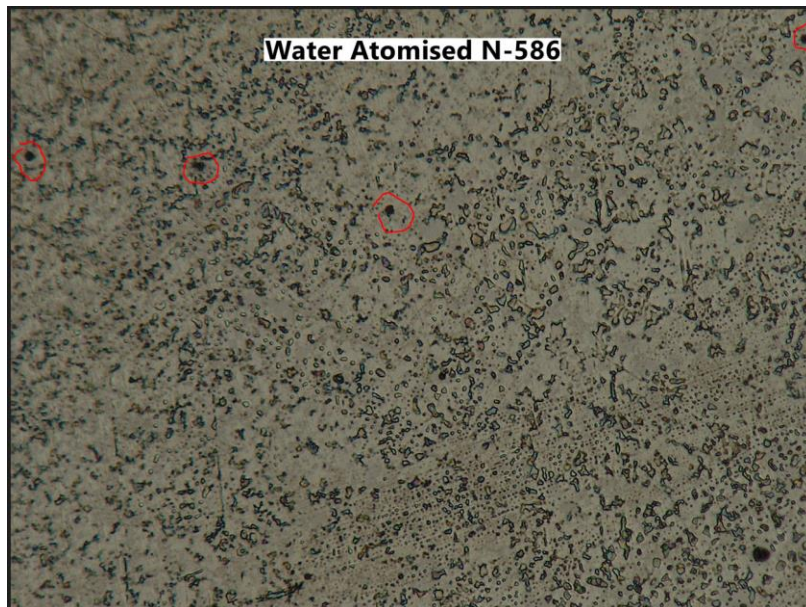


Figure 8:4. Metallography Grain Structure Image Deposition (x1000) Water Material

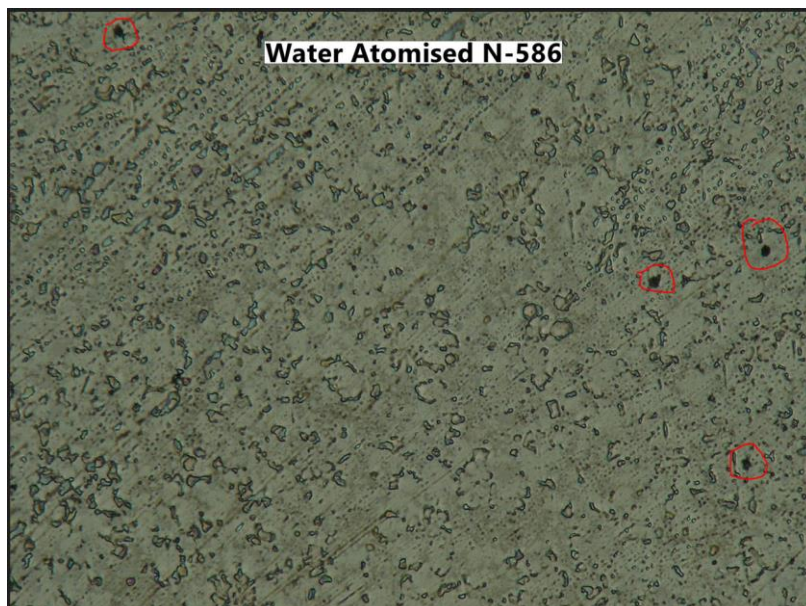


Figure 8:5. Metallography Grain Structure Image Deposition (x1000) Water Material

8.4 Deposition Porosity Count Comparison of Hybrid and Water Product

Images of both etched and non-etched were taken and a physical count conducted on both deposited mild-steel test pieces. From these metallography images it appears that both atomizing methodologies contain not too dissimilar degrees of porosity or voids of some kind although, the images do suggest one or two less holes in the water atomized product in this particular instance.

8.5 Deposition Conclusion / Findings for Both Atomizing Methodologies

As mentioned, deposited samples from both atomizing methodologies had similar counts of porosity/voids when evaluated with the images taken from the Keyence high-powered microscope. However, these specific counts were conducted on a single test piece whereby the test piece was ground and polished at a random area. What cannot be determined at this point is, however, is whether or not the porosity observed on these both deposited samples are actually gas porosity or, inter-dendritic porosity. Although, both types of porosity can be considered detrimental to the integrity of finished coating therefore, for determining particular type of porosity further, samples may need to be subjected to evaluation using SEM imaging equipment (scanning electron microscope). Using SEM may quantify more accurately the count of voids within the material coatings of both atomizing methodologies. Many deposited samples of both atomizing methodologies may, however, have to be deposited and evaluated further in order, to fully quantify the extent of type of porosity found rather than in this instance, a single test specimen. It is important to note however, that this specific weld deposit conducted on a small 40mm x 40mm x 10mm mild steel test piece attempts only to illustrate the “difference/comparison” in regards, to porosity in the material coating of both atomizing methodologies. This does not indicate the level of porosity which “may” be found in the finished coating upon the customers application.

9 Atomizing Energy Costs Comparison and Evaluation

Energy costs for both atomizing methodologies differ significantly. Hybrid atomizing consist of nitrogen and power (electricity) meanwhile, water atomized material consists mainly of power costs although, nitrogen is used for atomizing purging in this instance. These atomizing energy costs for both products especially now in recent times are considered to be a major expenditure which, is secondary to the metal costs itself.

9.1 “Current” Production Costs and Customer Demand for N-586 Material

Demand for this specific material grade exceeds ~eighteen thousand metric kg per annum. Although, demand does vary periodically. Table 9-1, shows the overall melts needed to be produced to meet current demand taking to product yield into account and also the optimum atomizing parameters which, are detailed within the manufacturing procedure. This table shows that approximately thirty percent less melts are required when product is produced by water methodology (~214 melts compared to ~303 melts). This equates to approximately Thirty-one metric tonnes of less material that needs to be assemble via the charge assembly department and subsequently melted. The water atomizing cycle time between melts is also approximate five minutes quicker when producing water atomized material.

Table 9-1. Atomizing Parameter & Demand Requirements (Hybrid v Water)

Atomising Parameters & Demand Requirements (Hybrid v Water):		
Alloy:	N-586X4-LC53	N-586X4-LC53
Atomising Medium:	Hybrid	Water
~Annual Sales Requirement (kgs)	18000	18000
~Product Yield (%)	17	24
Melt Qty (kgs)	105882	75000
Melt Size (kgs)	350	350
~Number of Melts Required	303	214
Atomizing Configuration	Fine Insert (Tapered)	25 / 8
Atomizing Pressure	300 psi	200 bar
~Atomizing Melt Tundish Pour time (mins)	16	16
Atomizing Nozzle Diameter (mm)	6	6
~Melt Cycle time (mins)	70	65

9.2 “Current” Production Gas Costs for Hybrid and Water Atomised Material

Both atomizing methodologies require nitrogen to produce material although, as per Table 9-2, suggests, water produced material does not consume any nitrogen gas at the atomizing head assembly itself. This is the only process area where, atomizing gas can be saved.

9.3 Hybrid Atomized Material Gas Costs

Approximately 647 Nm³ of nitrogen gas is required for both atomizing and purging purposes to produce hybrid material. Therefore, at a current atomizing unit nitrogen gas cost of £0.26 per Nm³ it cost approximately £168.20 to produce a single melt.

9.4 Water Atomized Material Gas Costs

Likewise, with water atomized material, only 390 Nm³ of nitrogen gas is required. This gas is required in this instance is for the use of purging purposes only. Therefore, at a current atomizing unit nitrogen gas cost of £0.26 per Nm³ it cost approximately £101.40 to produce a single melt.

Table 9-2. Atomizing Methodology Nitrogen Gas Costs (Hybrid v Water)

Atomizing Methodology Nitrogen Gas Costs (Hybrid v Water):		
Alloy:	N-586X4-LC53	N-586X4-LC53
Atomising Medium:	Hybrid	Water
Average Atomising Gas Consumption per 350kg Melt (Nm ³)	257	0
Average Purge / Drying Gas per 350kg Melt (Nm ³)	390	390
Total Nitrogen Gas Consumed per 350kg Melt (Nm ³)	647	390
Unit Cost of Nitrogen Gas (£/Nm ³)	0.26	0.26
Actual Atomizing Gas Costs (£)	66.8	0
Actual Purge / Drying Gas Costs (£)	101.4	101.4
Total Cost of Nitrogen Gas per Melt (£)	168.2	101.4

9.5 Gas Costs Comparison and Potential Savings

Between both methodologies, approximately £67 of nitrogen gas is saved per melt when producing water atomized material to that of the hybrid material. With the current finished product demand of 18,000 tonnes, and also with the reduced number of melts in regards, to the product being of a higher

yield in the water atomized product, the nitrogen gas saving therefore, in this instance would be in excess of £29 k per annum.

9.6 Current Production Electricity Costs for Hybrid and Water Atomised Material

Both atomizing methodologies require electricity via induction furnaces to produce material although, as per Table 9-3, suggests, water produced material consumes less electrical energy due to a lower cycle time. This lower melt cycle consists mainly of a lower tank settling time whereby a few minutes can be saved. This is the only process area where, electricity cost can be reduced between both the atomizing methodologies.

9.7 Hybrid Atomized Material Electricity Costs

Approximately 438 kw/hrs of electrical energy are consumed to produce a melt of hybrid material with a cycle time of 70 mins. Therefore, at a current atomizing unit electrical cost of ~£0.15 per kw hour, it cost approximately £66 to produce a single melt.

9.8 Water Atomized Material Electricity Costs

Approximately 406 kw/hrs of electrical energy are consumed to produce a melt of hybrid material with a cycle time of 65 mins. Therefore, at a current atomizing unit electrical cost of ~£0.15 per kw hour, it cost approximately £61 to produce a single melt.

Table 9-3. Atomizing Methodology Electricity Power Costs (Hybrid v Water)

Atomizing Methodology Electricity Power Costs (Hybrid v Water):		
Alloy:	N-586X4-LC53	N-586X4-LC53
Atomising Medium:	Hybrid	Water
Induction Furnace Power Capacity (kw/hr)	375	375
Induction Furnace Current (Amps)	550	550
Melt Process Cycle / Manufacturing Time (Mins)	70	65
KW Consumed @ 375 kw/hr	438	406
~Cost per kw/Hr	0.15	0.15
Total Cost of Power per Melt (£)	65.6	60.9

9.9 Electricity Costs Comparison and Potential Savings

Between both methodologies, approximately £5 of electrical energy is saved per melt when producing water atomized material to that of the hybrid material. With the current finished product demand of 18,000 tonnes, and also with the reduced number of melts in regards, to the product being of a higher yield in the water atomized product, the total electrical energy saving therefore, in this instance would be in excess of £6.8 k per annum.

9.10 Total Production Energy Costs for Both Hybrid and Water Atomized Product

The total difference in manufacturing energy costs between both atomizing methodologies is shown in Table 9-4. From data generated throughout this project the manufacturing costs have nearly halved for the production of this particular alloy alone. Total energy costs for hybrid methodology stands at ~£70 k per annum verses ~£35 k per annum for water atomized methodology. This figure takes in consideration yield improvement thus less melts required. Both these yield improvements and, using the cheaper water as an atomizing source, therefore, reduces the cost of producing this type of alloy by nearly 50%.

Table 9-4. Total Atomizing Energy & Annual Melt Requirement Costs (Hybrid v Water)

Total Atomizing Energy Costs £'s: (Nitrogen Gas + Power)		Annual Melt Requirement Cost (£)
Total Hybrid Atomizing Energy Costs per 350kg Melt (£)	233.8	70743
Total Water Atomizing Energy Costs per 350kg Melt (£)	162.3	34787

9.11 Conclusion of Energy Costs of Both Atomizing Mediums

Data from this research clearly show significant energy gas savings which, benefits the organization financially. It appears that by switching from gas to water saves the business approximately fifty percent of the energy costs, therefore, can remain profitable when producing this particular type of product. It is important to note however, although electrical cost per unit virtually remains the same regardless of atomizing methodology, the yield increase by the water method results in much fewer melts thus in principle saves unnecessary costly melts.

10 Dissertation Conclusion

This dissertation attempts to justify, by the research done why, this particular grade of material can be converted from hybrid to the water atomized methodology with no detriment to customers application or any ramifications to the overlay/coating. For this specific application, the primary function of the thermal spray coating is to reduce the wear rate on the pump shafts thus prolongs the service life of the part.

For similar type applications, water atomized material is generally the material used by many industries due to the cheaper conversion and energy production costs. However, on occasions hybrid produced material can be offered although, a financial premium which, is challenging to pass on to the customer especially, if the organization is trying to win over a customer contract from another similar material supplier.

From the out-set of this particular customer enquiry it is vital the technical sales representatives understand and appreciate the customers final application and its intended use in the field before offering a type of product, or indeed, an equivalent. Margins are often compromised in an attempt to win over an order. It can also be helpful to attain the production methodology of the competitor's material in order, to offer a like for like product. In this instance, the supplier's material was examined, and it appeared that it was probably water atomized due to its higher oxygen content similar to that of LSN's water atomized product although, as mentioned the competitor's manufacturing process is not fully conclusive.

Most importantly however, the customer in this instance had no formal specification for this material to denote atomizing methodology. The customers application process is similar to that of what is used in Europe and eastern Asia whereby, water produced atomized material is applied/sprayed in large volumes.

Data suggest that the production cost for this product would virtually half with the higher yield and the reduced atomizing nitrogen gas usage. This would be a step in the right direction to remain profitable.

Further work needs to be done however, on the water atomizing head assembly in regards, to perfect/optimize parameters to further increase and reduce the quantity of reported hollow spheres. The study of hollow particles on water atomized material in the organization has been virtually non-existent and only now, is still in its infancy of how varying atomizing parameters can effect this phenomenon since LSN Diffusion was established nearly thirteen years ago. Optimizing atomizing parameters may also result in more spherical particles which, help the screening process, this may reduce the screening inefficiencies thus, resulting in more yield.

Meeting the internal specification in regards, to the composition, particle size distribution and also the physical properties of material must always be paramount regardless of the atomising medium used. In this particular study, all water atomized material produced to date do indeed mimics the gas hybrid material. Laboratory evaluation on all water atomized material produced to date have all been approved and subsequently certified.

Although, the material has been fully stamped/certified, how will the overlay/coating will perform in service in regards, to its longevity of wear resistance or corrosion on the pumps shaft is unknown. How will the hybrid gas and the water atomized material differ in the field must be established.

10.1 Financial Impact to the Organisation

Although, LSN Diffusion produces a type of “niche” product, there are more and more manufacturers of metal powders coming into existence therefore cost savings of any type are much welcomed. Overseas manufacturers especially, in areas with lower living costs can offset margins with lower labour and administration cost are now starting to compete therefore, it’s imperative we remain innovative in the marketplace. The impact from a financial perspective of such improvements can be of significance especially when in this particular and unstable period, energy costs are erratic therefore, as mentioned continuous improvements to off-set increasing production costs have never been so much-needed.

10.2 Customer Feedback

It can be often difficult to receive customer feedback unless a particular customer issue has arisen. However, recently one of our Portuguese sales representatives who this territory belongs to, has indicated the possibility of increased sales via routine visit in one of the application facilities. Although, increased production in these facilities may be solely due to cheaper operational production costs on this particular part of the world. To date, there has been no negative feedback on product shipped.

The finding in this dissertation have also led to discussions with senior management in regards, other similar materials currently being produced by hybrid gas method. These particular specifications are being reviewed.

Water atomized material is often used as previously detailed in many applications, however, the conversion for this material may end up not being successful in regards, to the longevity of the sprayed component when in service. Although, to date, no issue has been raised by the customer. The percentage of hollow particles in the water atomized material although, higher than in the gas hybrid material may contribute to the failing of the coated oil shafts therefore, a substantial claim in regards, to the replacement of parts may exist as a possibility. On the other hand, the hybrid (gas) atomized material may have been over-engineered for what the intended use of the final component needs to be.

10.3 Dissertation Evaluation / Critique

This dissertation demonstrates a wide-ranging understanding and holistic perspective of the chosen topic from all the collected data used within this particular study. The wealth of all the analysis detailed, key points indicates that the proposed change of atomizing methodology is based on a solid foundation.

Although, this dissertation offers as mentioned, a good overview, it must also be noted that particular areas of this research can be significantly improved. Such areas include the literature review whereby, more relevant/specific credible theories could be sourced be further exploration to support and

enhance the accuracy/demand of the data and ideology of such a manufacturing methodology change. By such further background reading, a different path of study may have been taken which, may have resulted in a more concise and meaningful dissertation conclusion.

The data in this research may also benefit from being more robust by using more sophisticated analytical techniques to express a deeper analysis especially in the specific energy cost savings area. This may provide a more meaningful insight with a better understanding of such potential financial savings.

The data in this dissertation study indicates that substantial saving can be made to the organization. However, what is not included in this reported data is the conversion financial costs of converting the raw material into metallic powder as well as the savings on reduced consumable costs, factory burden and the reduced labour cost (kgs produced per man-hour) by the reduce melts required to manufacture this particular product. This dissertation for sure lacks all these additional cost savings which, could have been included to give a more holistic overview of this chosen project.

11 References

Akhtar, S., Ali, A., Haider, A. and Farooque, M., 2012. Measurement of powder density. *Key Engineering Materials*, 510, pp.597-601.

Amin, S. and Panchal, H., 2016. A review on thermal spray coating processes. *transfer*, 2(4), pp.556-563.

Andersen, O., Waag, U., Schneider, L., Stephani, G. and Kieback, B., 2000. Novel metallic hollow sphere structures. *Advanced engineering materials*, 2(4), pp.192-195.

Ang, A.S.M. and Berndt, C.C., 2014. A review of testing methods for thermal spray coatings. *International Material Reviews*.

Asgarian, A., Tang, Z., Bussmann, M. and Chattopadhyay, K., 2020. Water atomisation of metal powders. *Powder Metallurgy*, 63(4), pp.288-299.

Beckers, D., Ellendt, N., Fritsching, U. and Uhlenwinkel, V., 2020. Impact of process flow conditions on particle morphology in metal powder production via gas atomization. *Advanced Powder Technology*, 31(1), pp.300-311.

Coccia, M., 2018. The Fishbone diagram to identify, systematize and analyse the sources of general-purpose Technologies. *Journal of Social and Administrative Sciences*, 4(4), pp.291-303.

Das, S., 1999. Metallographic sample preparation techniques.

Declaration, S., 2014. *Guidelines for the Preparation of a Dissertation* (Doctoral dissertation, Medical University of Graz).

Disman, D., Ali, M. and Barliana, M.S., 2017. The use of quantitative research method and statistical data analysis in dissertation: an evaluation study. *International Journal of Education*, 10(1), pp.46-52.

Do, D., 2017. *The Five Principles of Lean*. [Online] Available at: <https://theleanway.net/The-Five-Principles-of-Lean> [Accessed 23 Mar 2024].

Emami, M., Sadeghi, M.H. and Sarhan, A.A., 2013. Investigating the effects of liquid atomization and delivery parameters of minimum quantity lubrication on the grinding process of Al₂O₃ engineering ceramics. *Journal of Manufacturing Processes*, 15(3), pp.374-388.

Fauchais, P.L., Heberlein, J.V., Boulos, M.I., Fauchais, P.L., Heberlein, J.V. and Boulos, M.I., 2014. Industrial applications of thermal spraying technology. *Thermal Spray Fundamentals: From Powder to Part*, pp.1401-1566

Feather, D., 2012. How to write a research methodology for an undergraduate dissertation. *Huddersfield: University of Huddersfield*.

Fedina, T., Sundqvist, J., Powell, J. and Kaplan, A.F., 2020. A comparative study of water and gas atomized low alloy steel powders for additive manufacturing. *Additive Manufacturing*, 36, p.101675

Fritsching, U. and Uhlenwinkel, V., 2012. Hybrid gas atomization for powder production. *Powder Metallurgy*, pp.99-124.

Ghassemieh, E., Versteeg, H.K. and Acar, M., 2006. The effect of nozzle geometry on the flow characteristics of small water jets. *Proceedings of the Institution of Mechanical Engineers, Part C: Journal of Mechanical Engineering Science*, 220(12), pp.1739-1753.

Gray, J. and Tillack, G.R., 2001. X-ray imaging methods over the last 25 years—new advances and capabilities. In *AIP Conference Proceedings* (Vol. 557, No. 1, pp. 16-32). American Institute of Physics.

Hyland, K., 2004. Graduates' gratitude: The generic structure of dissertation acknowledgements. *English for Specific purposes*, 23(3), pp.303-324.

Jiang, X., Siamas, G.A., Jagus, K. and Karayiannis, T.G., 2010. Physical modelling and advanced simulations of gas–liquid two-phase jet flows in atomization and sprays. *Progress in energy and combustion science*, 36(2), pp.131-167.

Kassym, K. and Perveen, A., 2020. Atomization processes of metal powders for 3D printing. *Materials today: proceedings*, 26, pp.1727-1733.

Khalid, K., Abdullah, H.H. and Kumar M, D., 2012. Get along with quantitative research process. *International Journal of Research in Management*, 2(2), pp.15-29.

Lagutkin, S., Achelis, L., Sheikhaliev, S., Uhlenwinkel, V. and Srivastava, V., 2004. Atomization process for metal powder. *Materials Science and Engineering: A*, 383(1), pp.1-6.

Lavernia, E.J., Srivatsan, T.S. and Rangel, R.H., 1992. Atomization of alloy powders. *Atomization and Sprays*, 2(3).

Lawley, A. and Murphy, T.F., 2003. Metallography of powder metallurgy materials. *Materials characterization*, 51(5), pp.315-327.

Le, T. and Henein, H., 1996. Effect of nozzle size and position on gas atomization. *International Journal of Powder Metallurgy*, 32, pp.353-364.

Mandal, S., Sadeghianjahromi, A. and Wang, C.C., 2022. Experimental investigations on molten metal atomization techniques—A critical review. *Advanced Powder Technology*, 33(11), p.103809.

Mostafaei, A., Hilla, C., Stevens, E.L., Nandwana, P., Elliott, A.M. and Chmielus, M., 2018. Comparison of characterization methods for differently atomized nickel-based alloy 625 powders. *Powder technology*, 333, pp.180-192.

Nouri, A. and Sola, A., 2019. Powder morphology in thermal spraying. *Journal of Advanced Manufacturing and Processing*, 1(3), p.e10020.

Pengjun, F., Yi, X., Xinggang, L. and Ya, C., 2018. Influence of atomizing gas and cooling rate on solidification characterization of nickel-based superalloy powders. *Rare Metal Materials and Engineering*, 47(2), pp.423-430.

Persson, F., 2021. *A Study of Parameters and Properties Influencing the Size, Morphology and Oxygen Content of Water Atomized Metal Powders* (KTH Royal Institute of Technology).

Radzikowska, J., 2000. Colour etching in foundry metallography. *Advanced materials & processes*, 158(1), p.31.

Radziwill, N., 2017. Creating ishikawa diagrams with R. *Software Quality Professional*, 20(1), pp.47-48.

Randolph, J., 2019. A guide to writing the dissertation literature review. *Practical assessment, research, and evaluation*, 14(1), p.13.

Ren, Z., Zhu, S., Wang, X., Zhao, Y., Han, G., Zhou, K., Wang, W. and Tian, G., 2023. Preparation and microstructure of multi-component high entropy alloy materials fabricated by gas atomization method. *Metals*, 13(2), p.432.

Pasupathy, M., Martín, J.M., Rivas, A., Iturriza, I. and Castro, F., 2016. Effect of the solidification time, rate on the median particle size of powders produced by water atomisation. *Powder Metallurgy*, 59(2), pp.128-141.

Persson, F., Eliasson, A. and Jönsson, P., 2012. Prediction of particle size for water atomised metal powders: parameter study. *Powder Metallurgy*, 55(1), pp.45-53.

Schade, C. and Dunkley, J.J., 2015. Atomization.

Schutt, R. (1996) *Investigating Social Research*. London: Sage Publications Ltd

Smith, R. and Hawkins, B., 2004. *Lean maintenance: reduce costs, improve quality, and increase market share*. Elsevier.

Sobolev, V.V., Guilemany, J.M. and Martin, A.J., 1995. Powder particle structure formation during solidification in the process of atomization. *Journal of materials processing technology*, 54(1-4), pp.249-260

Sobolev, V.V., Guilemany, J.M. and Martin, A.J., 1996. Formation of metallic powder particles during thermal interaction of liquid and solidified drops in the process of atomization. *Journal of materials processing technology*, 62(1-3), pp.216-224.

Soong, S.Z., Lai, W.L. and Kay Lup, A.N., 2023. Atomization of metallic alloy powders: Processes, parameters, and properties. *AIChE Journal*, 69(11), p.e18217.

Stankevich, A., 2017. Explaining the consumer decision-making process: Critical literature review. *The journal of international business research and marketing*, 2(6).

Sypeck, D.J., Parrish, P.A. and Wadley, H.N., 1998. Novel hollow powder porous structures. *MRS Online Proceedings Library*, 521, pp.205-210.

Talib, R.J., Saad, S., Toff, M.R.M. and Hashim, H., 2003. Thermal spray coating: A review. *Solid State Sci Technol*, 11(1), pp.109-117.

Thompson, B., 1988. Common methodology mistakes in dissertations: Improving dissertation quality.

Traina, K., Cloots, R., Bontempi, S., Lumay, G., Vandewalle, N. and Boschini, F., 2013. Flow abilities of powders and granular materials evidenced from dynamical tap density measurement. *Powder technology*, 235, pp.842-852.

Wang, G., Deng, Y., Adjei-Kyeremeh, F., Zhang, J., Raffeis, I., Bührig-Polaczek, A., Kaletsch, A. and Broeckmann, C., 2023. Numerical analysis of droplet breakup, cooling, and solidification of gas atomisation. *Powder Metallurgy*, 66(5), pp.493-508.

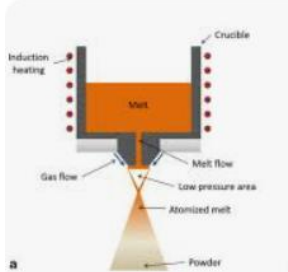
Yenwiset, S. and Yenwiset, T., 2016. The effect of the molten metal stream's shape on particle size distribution of water atomized metal powders. *Engineering Journal*, 20(1), pp.187-197.

Qinyang Li, Yu, Jinku, Yuehua Wang, XiCan Zhao, Qi Qiao, Jia Zhao, and Sen Zhai. "Wear resistance of Ni-based metallic alloy coatings." *Advances in Materials Science and Engineering* (2019).

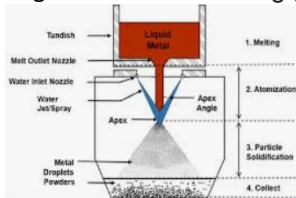
Zhong, W. et al., 2023. Comparative Study of Nickel-Based Powders Atomized by Argon and Nitrogen. *Powder Metallurgy and Metal Ceramics*, Volume Vol.,61, pp. 633-643.

12 Endnotes

ⁱ Atomization is a process in this instance whereby, a molten metal stream is broken into small droplets (metallic powder). Nitrogen can be used as the atomizing medium (*Schade, C. and Dunkley, J.J., 2015*).



ⁱⁱ Water atomization uses water as the atomizing medium which, is considered as cheaper alternative than that of gas atomization Soong (*S.Z., Lai, W.L. and Kay Lup, A.N., 2023*).



ⁱⁱⁱ Energy costs both electricity & gas (nitrogen) have risen sharply by over 50% from late 2022. The cost of electricity has risen by nearly 50% The price of nitrogen atomizing gas has doubled in unit cost.

^{iv} Hybrid atomization is a process whereby the gas atomized material is collected in water at the bottom of the atomizing chamber which, is subsequently dried in nitrogen hot-oil dryers (*Fritsching, U. and Uhlenwinkel, V., 2012*).

^v Spraying application on pump shafts used by the customer for the alloy N-586



^{vi} The packing density is the relationship/ratio of a solid volume to the total volume of the metallic powder sample. This packing density can be affected by its morphology and also its particle size which, can also determine the physical flow of the material through a calibrated orifice (*Akhtar, S., Ali, A., Haider, A. and Farooque, M., 2012*).

vii Hall Cup flowrate is the time taken of a specified mass of material to flow through the orifice of a Hall Flowmeter under gravity which, is specified in particular standards (*Akhtar, S., Ali, A., Haider, A. and Farooque, M., 2012*).



viii HVOF application is a type of thermal spray coating process that, involves propelling metallic powder at high speeds onto a substrate/base metal which, can create a dense and tightly bonded overlay.

ix Plasma spraying metallic powders are a type of thermal spray coating whereby, material is melted in the plasma arc and subsequently propelled onto a base-metal which, is used in many industrial industries.

x Secondary satelliting of dry gas atomizing can occur when the finer atomized metallic powder recirculates back-up into the atomizing chamber and re-attaches to the particles which, have not yet solidified/frozen.

xi Tap and apparent density involves the measuring the bulk density of a particular material under a controlled condition. It is performed by calculating its mass by filling a known volume. Density is one of the characterization tests of a material which, aims to evaluate flow and packing efficiency (*Traina, K., & Cloots, R, et al, 2013*).

xii Morphology of metallic powders can refer to the physical characteristics of the material such as shape, surface roughness and other surface features. Understanding these types of characteristics can be crucial for the optimization of parameters to produce desired properties (*Beckers, D., Ellendt, N. et al, 2020*).

xiii A powder sample riffler is a device which, is used to split a powder sample into smaller equal proportions. Ideology of a powder sampler is to ensure a fair representation in regards, to the homogeneity of the original sample