



# Inspiring Great British Manufacturing

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## Executive summary

This executive summary covers all activity conducted in the first evaluation campaign, WP 3000, of the “ESA Additive Manufacturing Powder Material Supply Chain: Verification and Validation” project (ITT reference: G61A-018QT).

In WP 3100, four AlSi10Mg powders purchased from different suppliers (in WP 2000) were sampled and dispatched to three AM bureaus, and multiple powder characterisation test providers. Two of the four suppliers provided the minimum requirement of information on their Certificate of Conformance (CoC) whilst 2 other provided more than the minimum requirement. Two of the powders suppliers also did not meet all the size specifications included on the specification, as determined by their own evaluations. Powder samples for the characterisation laboratories were taken from the master batch via keystone sampler due to the hazardous rating of AlSi10Mg powder. This was performed in a temperature and humidity controlled environment. Learning from this activity has been summarised in TN 3.1.

In WP 3200, powder characterisation tests were performed at consortium laboratories and external contract laboratories. Both well-established and novel powder characterisation tests were performed. The results of the powder characterisation tests were reported, and the tests and laboratories were evaluated for repeatability, reproducibility and consistency (where appropriate). From consortium testing, all powder batches passed the specification criteria for chemistry, morphology, tapped density and apparent density. However two powders failed on size evaluations and one failed on flow performance as evaluated by funnel flow. Again considering consortium tests, size, density and thermal evaluations were found to be repeatable and reproducible. Shape evaluations were generally repeatable, however exhibited low reproducibility. Rheological evaluations of powders, as evaluated by funnel flow tests, Dynamic Angle of Repose test, and rheometer and shear cell tests were considered to have low repeatability and reproducibility. Size evaluations proved to be inconsistent across test techniques. This was both when comparing actual values and trends between coarsest to finest powders. Whilst the differences in values could be explained by virtue of the calculation and conditions used in the tests, comparative analyses still did not always provide comparable results. Shape evaluations provided more consistent values between tests than size evaluations, as the trends between shape analyses remained consistent. Bulk density measurements were shown to be the most consistent. In a variety of test conditions, bulk density measurements provided similar values and therefore the same trends between powders. This is detailed in TN 3.2 Chapter 2 and 3.

Novel powder characterisation tests were conducted at external contract laboratories. X-ray Computed Tomography and automated-SEM were found to be immature evaluation techniques, that were dependant on the skill of the operator and lacked a standardised process. They were evaluated to have low reproducibility. Dynamic Vapour Sorption evaluations were close to the detection limit of the machine and provided information that was not relevant to a powder specification. The specific surface area evaluation provided by BET analysis was found to correlate with the morphology of the powders. Gas pycnometry determined differences between the porosity of powders (internal) well. Inspire’s spreading testbed evaluation of layer density correlated well with consortium packing density evaluations. The GranuDrum evaluations did not correlate with, or have the same test procedure or data evaluation methodology, as a similar Dynamic Angle of Repose

test (Revolution). This called into question the relevance of these tests. The GranuTap test correlated well with packing evaluation made by the consortium. The GranuCharge was deemed to be very sensitive to the operator, but did correlate with the spreading test best evaluation from Sirris, when evaluating the effect of blade material. This is detailed in TN 3.2 Chapter 4.

Correlations between powder properties were investigated, and statistical analysis was performed to ascertain correlations between inherent powder properties (size, shape and oxygen content) and powder performance evaluations (density and flow). Comparing all rheological tests which claimed to evaluate the same properties of flow, there were no clear agreements of actual values or trends. Indeed, the flow characteristics of the powders changed depending on the test examined. This highlighted the impact of the test condition on the measured flow behaviour of the powder. In the univariate analysis, shape correlations were found to have the greatest number of correlations to powder performance properties (compared to size). Correlations between bulk density performance parameters and size and shape parameters were found in multiple tests, and were consistent across tests. Rheological test data did not exhibit the same correlations between intrinsic powder properties and rheological properties across multiple tests. In the multivariate analysis, it was shown that the combination of size and shape parameters correlated with density measurements better than size or shape alone. Including the oxygen content of materials within the multivariate data analysis reduced the correlations found between intrinsic powder properties and performance powder properties, which indicated that oxygen content was unlikely to drive performance behaviour. This analysis suggested that AM users could have confidence that the tests that are originally included on the specification document are robust enough to provide a consistent pass or fail result. This is detailed in TN 3.2 Chapter 4 and 5.

Concurrently in WP 3300, benchmarking artefacts and representative space parts were manufactured via AM. Manufactured components were then extensively characterised using both destructive and non-destructive analyses and the results reported. The characterizations on the AM test artefacts provided a substantial amount of data on the quality and properties of the parts. There were no build failures and all parts were successfully built. The 12 builds produced 39 artefacts each (totalling 468 artefacts). Evaluations included data from all bureaus so to gain an appreciation of the impact of the AM processing route compared to the impact of the powder. The investigations showed that the AM process has a major influence on the properties of the printed AlSi10Mg parts and whilst the characteristics of the powder material were reflected in the part characteristics, it was mostly in a secondary manner. The shape accuracy of the parts were clearly connected to the AM process, but were also influenced by the powder properties. Similarly, the surface roughness of the parts were influenced both by the AM process (including post-processing) and the powder. It is proposed that the shape of the melt pools in the microstructure, and the used parameters for contouring and the bulk influenced the topography of the surface areas. Mechanical properties could also be connected both to the AM process and the powder. A low standard deviation on mechanical properties was connected to a homogeneous microstructure free from large defects. Presence of large defects lowered the ultimate tensile strength and elongation values and caused scatter for the results between different bars. Heat treatment reduced the scatter. Un-treated materials are most sensitive to display the eventual presence of large defects within a batch. The defects in the tensile bars originated from disturbances in the powder spreading behaviour. This is detailed in TN 3.3.

In WP 3400, the results of the powder characterisation tests and part evaluations were cross-examined so to investigate which powder properties influence on specific part properties. Of the lab-based flow characterisation tests, the spreading testbed at Inspire best replicated in-process powder spreading behaviour. Evaluations of powder triboelectric charging and its effect on spreading behaviour were not consistent with in-process spreading behaviour. Also, there was no clear link between a slightly coarser PSD (away from the nominal), or the presence of rare oversized agglomerates and the porosity of final parts. Lab-based and in-process density measurements correlate well across most tests, where poured and apparent density correlate most with in-process spreading behaviour. There was more of a correlation between particle shape and in-process spreading performance than there was with particle size. When evaluating part density, the AM process could tolerate a higher level of variation of in-process layer density than powder porosity, such that the effect of particle porosity was more dominant than in-process layer density on part porosity. In this evaluation, there was no clear link between shape accuracy of large features and in-process spreading behaviour. This is detailed in TN 3.4.

The learning from this activity allowed the modification of the original powder procurement specification, so to focus on the powder properties with the most influence on part properties, and to better define those properties in-line with the values witnessed in the first round. The particle size distribution descriptors were removed (as was the sieve analysis), and only undersized and oversized limits were included. Also the BET specific surface area was added. Both these changes were made to reflect the proposed greater importance of the shape evaluations. Funnel flow evaluation was removed, and the oxygen and hydrogen contents specifications were modified. Gas pycnometry was also added to the specification. This is detailed in PS 2.

For powder characterisation in WP 4000, test evaluations were then re-selected based on the tests included in the specification, and on the importance of the evaluated property as defined in WP 3200. Tests to be conducted in WP 4000 are: ASEM, Dynamic Angle of Repose (GranuDrum and Revolution), poured, apparent and tapped density, SEM (for imaging morphology), dynamic Image Analysis, laser diffraction, chemistry (ICP and ONH), layer density evaluation, laser absorptivity, GranuTap, helium pycnometry, Karl-Fischer titration moisture analysis, BET and analysis by a new machine to the market, the Microtrac Sync.

The selection of powder suppliers for work in WP 4000 was originally intended to be based on the suppliers ability to respond to the PS2 specification. This updated specification was then sent to suppliers for comment, however there was limited engagement from the supply chain. Due to this, suppliers were then chosen based on their standard AlSi10Mg product offerings, where the most ideal powder properties were targeted, alongside any potential new learnings for the project. Tekna and GE (AP&C) were the chosen suppliers for WP 4000 activities.

Part evaluations in WP 4000 are recommended to focus on the characterization of mechanical properties in as-build and machined bars as tensile testing reflects the presence of large defect and spreadability of the powder. The process observations provided by the AM bureaus was valuable input for the analysis of the results. It is recommended that the selection of bureaus in WP 4000 will be based on fluency of communication between the customer and bureau and on an ability to provide fine surfaces being advantageous for tensile testing of as-build bars. This is discussed in TN 3.4.

**Version Control**

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