



D4.2. Datasheet of thermophysical and mechanical properties of light metal alloys by WAAM

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TABLE OF CONTENTS

| T | ABLE O | F CONTENTS | 2 |
|---|--------|---|----|
| P | ROJECT | INFORMATION | 3 |
| | | ABLE DETAILS | |
| | | oduction | |
| | 1.1 | Alloy selection and WAAM process parameters | |
| | | Test matrix | |
| | | ults | |
| | 2.1 | Tensile testing at room temperature and 77K | |
| | 2.2 | Light optical microscopy | |
| | 2.3 | Scanning electron microscopy | |
| | 2.4 | Material properties for FEM analysis | 10 |
| 3 | Sum | nmary and Outlook | |





PROJECT INFORMATION

<u>Project full title</u>: Maturing the production standards of ultraporous structures for high density hydrogen storage bank operating on swinging temperatures and low compression

Acronym: MAST3RBoost

Call: HORIZON-CL4-2021-RESILIENCE-01

Topic: HORIZON-CL4-2021-RESILIENCE-01-17

Start date: 1st June 2022

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List of participants:

| Number | Name of beneficiary | Acronym of beneficiary | Country |
|--------|---|------------------------|--------------|
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| 6 | Nanolayers | NANO | Estonia |
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| 9 | University of Pretoria | UP | South Africa |
| 10 | Council for Scientific and Industrial Research | CSIR | South Africa |
| 11 | PSA | PSA | Portugal |
| 12 | TWI Ltd | TWI | UK |
| 13 | University of Nottingham | UoN | UK |





DELIVERABLE DETAILS

| Document Number: | D4.2 |
|-------------------------|--|
| Document Title: | Datasheet of thermo-physical and mechanical properties of light metal alloys by WAAM |
| Dissemination level | PU – Public |
| Period: | PR2 |
| WP: | WP4 – "Development and testing of advanced, fibre-reinforced lightweight metal alloys and coatings for hydrogen storage vessel" |
| Task: | 4.1 – "Development of a catalogue of lightweight metal alloys and preliminary testing of sample specimens based on waDED state-of-the-art". |
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| Abstract: | Deliverable D4.2 is designed to be a preliminary summary of all mechanical and metallographic characterization performed on WAAM samples in this work package so far. Three specific materials, as defined in the original project proposal, were selected for preliminary testing. Two medium to high strength aluminium alloys, i.e. MA-5183 and MA-6063, and one high strength magnesium alloy AZ91 were chosen. This preliminary data |
| | sheet includes results from tensile testing (room temperature and 77K), light optical microscopy and scanning electron microscopy. |





1 Introduction

This document contains the deliverable 4.2, "Datasheet of thermophysical and mechanical properties of light metals alloys by WAAM" associated with the task 4.1 "Development of a catalogue of lightweight metal alloys and preliminary testing of sample specimens based on waDED state-of-the-art" [M1-M18] and the WP4 "Development and testing of advanced, fibre-reinforced lightweight metal alloys and coatings for hydrogen storage vessel" [M1-M36].

Deliverable D4.2 is designed to be a preliminary summary of all mechanical and metallographic characterization performed on WAAM samples in this work package so far. During the course of the literature study (see D4.1), which included a thorough review of industry standards and market availability, three specific materials, as defined in the original project proposal, were selected for preliminary testing.

Two medium to high strength aluminium alloys, i.e. MA-5183 and MA-6063, and one high strength magnesium alloy AZ91 were chosen. The project proposal also mentions investigations into composite materials produced via WAAM, i.e. carbon fibre reinforced aluminium alloys, but this material group is not included in this report. The TRL level of the proposed WAAM adaptation is still too low at this point of the project to produce sufficient samples for testing and characterization. Depending on the progress of this task (T4.2), testing data will be provided in D4.3.

1.1 Alloy selection and WAAM process parameters

Based on the findings of the literature survey and prior experience the non-heat treatable aluminium alloy AlMg4.5Mn0.7 (MA-5183) was chosen as the primary candidate as WAAM feedstock material. It combines medium strength and ductility levels with good weldability without the need of heat treatment. Due to the size and complexity of the vessel design water quenching (which is necessary for heat treatment) would be technologically challenging and would also result in significant unwanted distortion.

Higher strength levels are only achievable by aforementioned problematic heat treatment; for comparison and as additional benchmark the heat treatable aluminium alloy AIMg0.7SiTiB (MA-6063) was included in the test matrix. It needs to be evaluated on a case-by-case basis if the increase in strength justifies the increase in complexity during manufacturing.

A further reduction in vessel weight could be achieved by using magnesium as construction material. Therefore, a high strength Mg alloy, AZ91 was added to the test matrix.

1.2 Test matrix

A test matrix consisting of tensile testing at room temperature (RT) and cryogenic conditions (77K), light optical microscopy (LOM) and scanning electron microscopy (SEM) including energy dispersive x-ray analysis (EDX) was set up for the three selected alloys (Figure 1).

Testing and characterization of MA-5183 specimens was conducted by LKR (coordinated with TWI). MA-6063 results are obtained from literature, suitable studies are available and thereby testing effort could be minimized. AZ91 results were obtained by LKR.





| Task | MA-5183 | MA-6063 | AZ91 |
|--|----------|----------|----------|
| Tensile testing at room temperature (RT) | √ | √ | √ |
| Cryogenic tensile testing (77K) | √ | | ✓ |
| LOM | √ | √ | |
| SEM/EDX | √ | | |

Figure 1: Test matrix for the three selected construction materials.

2 RESULTS

2.1 Tensile testing at room temperature and 77K

Tensile testing of MA-5183 and AZ91 was performed at standard conditions (RT) and at 77K, which is the minimum operating temperature of the MAST3RBoost vessel. Results for MA-6063 are only available at RT.

It is well known that tensile strength of metals increases with decreasing testing temperatures. Most of the time this strength increase is coupled with a decrease in ductility, however for some face-centered-cubic (fcc) structured metals, such as aluminium the ductility is not impaired and can be even slightly increased. For wrought aluminium alloys this effect is well documented, but for WAAM material no literature data is available. The lattice structure of the magnesium alloy AZ91 is hexagonal close-packed, entailing a ductility decrease at lower temperatures.

MA-5183 WAAM specimens in as-built condition (no heat treatment) show a distinct increase in yield (138 -> 164 MPa) and tensile strength (298 -> 433 MPa) from room temperature to 77K, accompanied with a slight increase in fracture elongation (28 -> 29%). The results are independent of the testing direction – isotropic, which is also an important criterion for WAAM samples (Figure 2). In comparison the yield strength (74 MPa), tensile strength (155 MPa) as well as fracture elongation (15%) of MA-6063 specimens in as-built condition at RT are significantly lower. Only by a two-step heat treatment (solution treatment at 540°C for 2h, water quenching and artificial ageing at 165°C for 13h), higher values compared to MA-5183 can be achieved (Figure 3). The AZ91 samples in as-built condition also show a strength increase from RT to 77K (YS: 125 MPa -> 154 MPa, UTS: 268 MPa -> 299 MPa), but at a quite low ductility level (9 -> 4%).

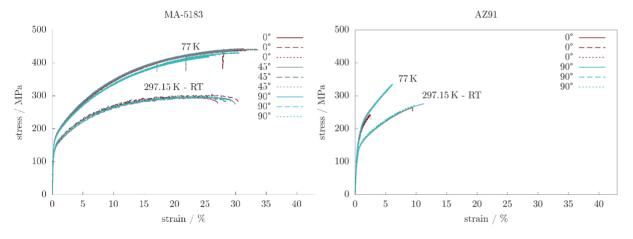


Figure 2: Stress strain curves for MA-5183 and AZ91 WAAM samples in vertical and horizontal testing direction.



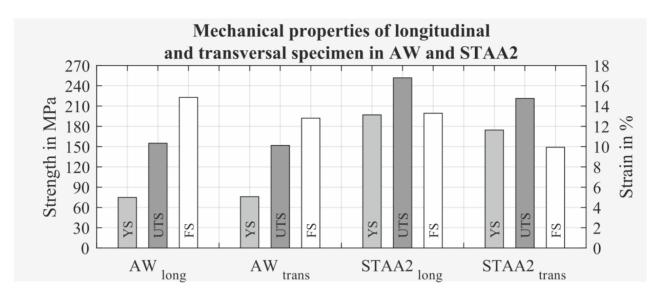


Figure 3: Summary of tensile properties of MA-6063 WAAM specimens [1].

2.2 Light optical microscopy

On the one hand light optical microscopy is used to qualitatively analyse porosity levels in the WAAM specimens. On the other hand, it visualises the grain structure, highlighting the desired isotropic grain morphology.

Unwanted pores are oftentimes an issue in aluminium alloys, especially in welded parts. Most pores are caused by a steep drop of hydrogen solubility from liquid to solid state. Limited amount of porosity, especially small pores, can be tolerated as it has limited influence on the mechanical properties of the samples. Hence low porosity levels are a quality indicator of different feedstock material.

The WAAM samples of MA-5183 show a very low number of pores throughout the complete cross sections. Also, the pore sizes are rather small, the majority are below 50 μ m (Figure 4).

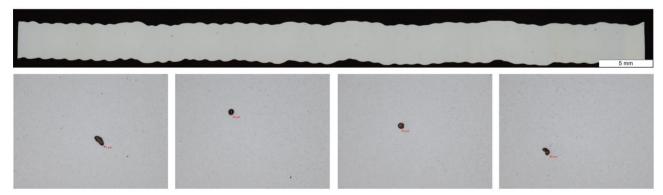


Figure 4: Polished cross sections of MA-5183 sample produced via WAAM.

In the MA-6063 cross sections the pore content and size distribution are higher but still acceptable for WAAM samples (Figure 5).

Porosity is not a big issue in magnesium alloys, hence AZ91 was not analyzed.







Figure 5: Unetched cross section of MA-6063 WAAM sample [1].

The etched cross sections of MA-5183 reveal equiaxed grains, homogeneously distributed in welding and build direction (Figure 6). The approximate grain size is in the range from 60-70 µm.

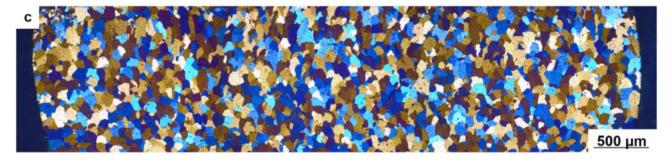


Figure 6: Etched (barker etchant) cross sections showing the equiaxed microstructure of MA-5183 WAAM material [2].

The grains in the MA-6063 are also equiaxed and homogenously distributed. The grain size, mostly independent of the heat treatment condition, ranges from 20-30 µm (Figure 7). The smaller grain size in 6063 is attributed to the usage of TiB as grain refining agent.





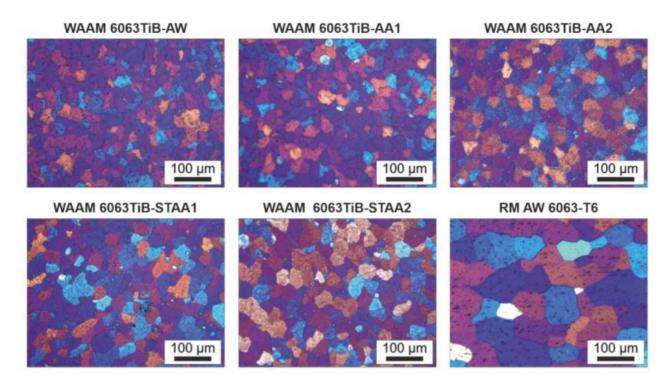


Figure 7: Microstructure of MA-6063 in different heat treatment conditions in comparison to the wrought base material AW 6063-T3 [1].

2.3 Scanning electron microscopy

Scanning electron microscopy (SEM) enables more detailed insights into microstructural features of the WAAM samples. SEM and EDX was only performed for the MA-5183 alloy.

Secondary electron (SE) mode imaging was combined with back scattered electron (BSE) mode and energy dispersive x-ray spectroscopy for phase identification. Mn- rich phases, most likely Al₆Mn intermetallic phases, were detected. Small pores, size range below 10 µm, are also visible.

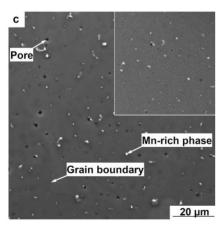


Figure 8: SEM image of MA-5183 WAAM specimens, SE mode with BSE inset [2].





2.4 Material properties for FEM analysis

The design and dimensions of the MAST3RBoost vessel will be checked and evaluated based on the ASME VIII Div. 2, which requires certain material properties as input data. Usually, these material properties are found in extensive databases as part of the standard (ASME B31.12), but for WAAM material no certified properties are available yet.

Table 1 summarizes measured material properties of MA-5183 WAAM material at 77K and 298K, which can be used for the FEM analysis according to ASME VIII Div. 2. The coefficient of thermal expansion and strain energy are not yet available and will be added to Deliverable D4.7.

Table 1: List of selected MA-5183 WAAM material properties at 77K and 298K.

| | 77K | 298K |
|-----------------------------|---------------|---------------|
| Young's modulus | 86.9±6.7 GPa | 69.6±3.2 GPa |
| Elastic limit | 164.2±2.1 MPa | 138.1±2.4 MPa |
| Coeff. of thermal expansion | tbm | tbm |
| Breaking limit | 433.4±8.0 MPa | 298.3±2.8 MPa |
| Elongation at break | 29.2±2.6 % | 28.3±1.1 % |
| Strain energy | tbm | tbm |

Tbm... to be measured

3 SUMMARY AND OUTLOOK

The data compiled in the preliminary data sheet strengthens and supports the choice of MA-5183 as primary construction material using the wire arc additive manufacturing technology for the MAST3RBoost vessel. It combines low density with robust isotropic strength values (in as-built condition!) and good weldability. (Small) equiaxed grains and low porosity also positively attribute to its property portfolio.

In comparison, MA-6063 showed weak mechanical properties (in as-built condition), which can only be improved by heat treatment. Due to its size and complexity, heat treatments including water quenching were ruled out by the WP4+WP5 consortium. Furthermore, the porosity content was visibly higher in this alloy.

Magnesium alloys are very promising for ultra-lightweight design, due to its excellent strength to weight ratio, but are limited by their low ductility, bad corrosion resistance and little available data on hydrogen embrittlement.

Based on these findings' further characterization work, e.g. fatigue and influence of hydrogen, in task 4.3 and 4.4 will be conducted using MA-5183 only. Some outstanding characterization work, e.g. CT measurements to numerically assess porosity and other welding related defects or chemical homogeneity of the WAAM samples will be reported in the final data sheets (D4.7). D4.2 combined with D4.7 will provide invaluable data on microstructural and mechanical properties of MA-5183 at cryogenic conditions under hydrogen atmosphere.





References

- [1] R. Winterkorn, A. Pittner, and M. Rethmeier, "metals Wire Arc Additive Manufacturing with Novel Al-Mg-Si Filler Wire-Assessment of Weld Quality and Mechanical Properties," 2021, doi: 10.3390/met11081243.
- [2] T. Klein and M. Schnall, "Control of macro-/microstructure and mechanical properties of a wire-arc additive manufactured aluminum alloy," *Int. J. Adv. Manuf. Technol.*, vol. 108, no. 1–2, pp. 235–244, 2020, doi: 10.1007/s00170-020-05396-6.