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Exmet AB c/o Swerim PO Box 7047, S-164 07 Kista Sweden	Project Manufacturing of Metallic Glasses for Bearing Applications ESA Contract No 4000123990/18/NL/LvH/zk
	Document Title Executive Summary – Confidential version

1 Introduction of the context

1.1 Scope

When the original project idea was conceived and discussed between Exmet AB, SKF AB, Rymdstyrelsen AB, and ESA, bearings for space applications were the long-term targeted products. At a later point, it was decided that the format should be shrunk to a De-risk Activity with a shorter-term target of evaluating the material rather than producing actual bearings.

1.2 Technical background and Objectives of this activity

Compared to *crystalline* metals, *amorphous* metal alloys have an outstanding combination of hardness, strength and elasticity. The improved mechanical properties could decrease life-cycle cost and decrease weight of vital spacecraft components. The lack of grain boundaries could give advantageous tribological surface conditions and thus decrease the need for warm lubrication.

The main technical objective of this activity was to build and evaluate additively manufactured parts with amorphous microstructure of an iron-based alloy, aiming for the following properties:

- Part dimension > 1 cm
- Relative density > 97%
- Structure > 80% amorphous
- Hardness > 900 HV

Previously, the applications of amorphous metal have been limited to very thin structures because very high cooling rates are needed to form an amorphous microstructure. The use of additive manufacturing (AM) overcomes this problem since the small melt pool results in a very high local cooling rate.

2 Description of work

2.1 Technical steps

In summary, the project was outlined as follows;

- Selecting Fe-based alloys with potential to fulfil the given requirements. Procuring alloy powders from an external provider of gas-atomised metal powder.
- Developing AM process parameters for the alloys. Evaluating the properties of the alloys.
- Selecting the most promising alloy. Manufacturing washers to be tested by SKF, aiming for their Multi-Contact Test (MCT) method. Evaluation.

2.2 Programme of work

In Table 1 the work packages (WPs) are listed in a Work Breakdown Structure (WBS).

WP no.	Title	Deliverables	Company	Manager
1	Alloy definition	HW1, D1	Exmet	Mattias Unosson
2	Material development	HW2, D2	Exmet	Lena Thorsson
3	Washer development	HW3, D3	Exmet	Lena Thorsson
4	Washer evaluation	D4	SKF	Marcel Escursell
5	Reporting	TDP, ESR, FR, CE, TAT & WAT	All	Mattias Unosson
6	Project management	CCD	Exmet	Mattias Unosson

Table 1. Work Breakdown Structure (WBS).

3 Activities performed

3.1 Alloy definition (WP1)

For the definition of alloy candidates, a combination of empirical evidence from previous SLM processing of iron alloys, computational thermodynamics and Inoue's three empirical rules¹ for designing bulk metallic glasses was used. The three compositions are listed in Table 2.

Alloy candidate designation	Composition (wt-%)
ESA 1	$\text{Fe}_{34}\text{Ni}_{30}\text{Cr}_{24}\text{Mo}_{10}\text{B}_1\text{C}_1$
ESA 2	$\text{Fe}_{63.46}\text{Ni}_{16.67}\text{Mo}_{9.6}\text{P}_{7.44}\text{C}_{2.4}\text{B}_{0.43}$
ESA 3	$\text{Fe}_{60.5}\text{Mo}_{18}\text{Ni}_{13}\text{Cr}_7\text{B}_{1.5}$

Table 2. Compositions of alloy candidates for the activity [CONFIDENTIAL].

3.2 Material development (WP2)

Each alloy was processed and evaluated in order to create solid metallic glass material samples. After adjusting processing parameters and comparing resulting material properties, the most promising material and corresponding process was selected for further work in the project. The utilised Additive Manufacturing (AM) process was Selective Laser Melting (SLM).

3.3 Washer development (WP3)

The alloy that showed the best bulk material properties in WP2 was selected for the remaining project activities. In WP3, SLM process parameters for this selected alloy ("ESA1") were further developed and optimized; then samples for evaluation and washers intended for testing in WP4 were built.

3.4 Washer evaluation (WP4)

The rolling contact fatigue performance of the washers was to be evaluated by Multi-Contact Test (MCT) in WP4. A critical factor to achieve good rolling contact fatigue for bearing applications is to ensure high density material without defects. The presence of pores, cracks and other defects in the material would drastically reduce the fatigue life of a bearing. In order to increase the density of the material and remove any cracks and pores, it was decided to treat the samples produced in WP3 with Hot Isostatic Pressing (HIP) before the MCT evaluation.

4 Main results

4.1 Alloy definition (WP1)

Main output of WP1 is the three procured alloy powders, and the data resulting from the characterization of said powders.

¹ Inoue, A. (2000). Stabilization of metallic supercooled liquid and bulk amorphous alloys. Acta materialia, 48(1), 279-306.

4.2 Material development (WP2)

When “best” laser parameters had been determined, samples intended for evaluation and the hardware delivery samples HW2 were built (shown in Figure 1 - Figure 3).



Figure 1. ESA1 LTH255.

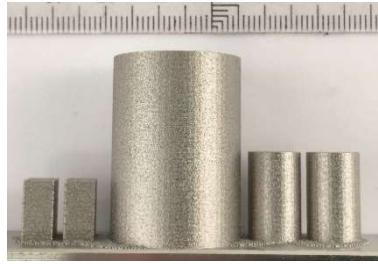


Figure 2. ESA2 LTH259.

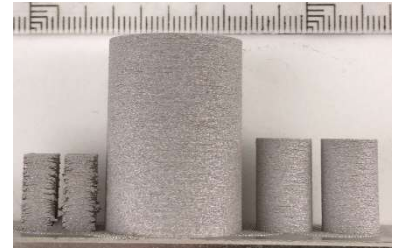


Figure 3. ESA3 LTH262.

Comparing the exterior appearances, it was determined that the ESA1 samples looked best; showing a consistent and matte surface for both cylindrical and square samples, without visible edge cracks that were especially apparent on the square corners of ESA2 and ESA3 samples.

4.2.1 Microstructure

Light optical microscopy (LOM) was used to document porosity and micro-cracking patterns in polished cross-cuts; best one of each alloy can be seen in Figure 4 - Figure 6. All scale bars = 400 μm .

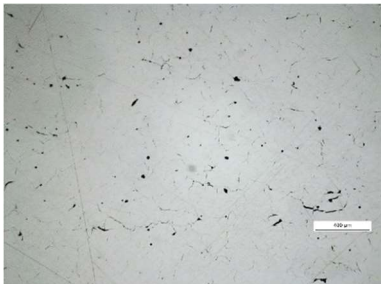


Figure 4. ESA1 LTH252-10.

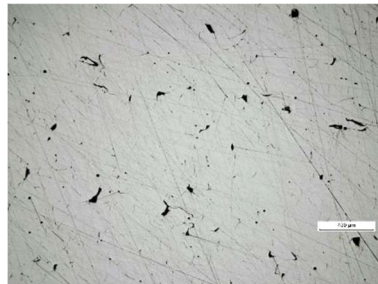


Figure 5. ESA2 LTH258-6.



Figure 6. ESA3 LTH261-1.

In total, the microstructure of SLM processed alloy ESA1 was deemed the most promising of the three candidates. It was observed that Alloy ESA2 was brittle and difficult to work with, as the material appeared to crumble during polishing. Edge cracks were present in all alloys, typically 200-300 μm deep in ESA1 and 2, but up to 1 mm deep in ESA3. Such edge cracks would weaken the mechanical properties and be difficult to remove by post-processing.

4.2.2 Hardness

Alloys were comparatively hard, as expected; ESA1 showed a hardness of 1000 HV and ESA2 830 HV. ESA3 was not evaluated since it had been de-selected as an alloy candidate.

4.2.3 XRD

X-ray diffraction (XRD) was used to analyse the amorphicity of the samples. Comparing the three curves in Figure 7, it is clear that alloy ESA2 (red curve) had the highest amorphous fraction as no distinct Bragg peaks appear. The indistinct Bragg peaks of ESA1 (green) and ESA3 (black) curves indicate some crystalline formations, but the interpretation of peaks with such broad bases are that any crystals must be small.

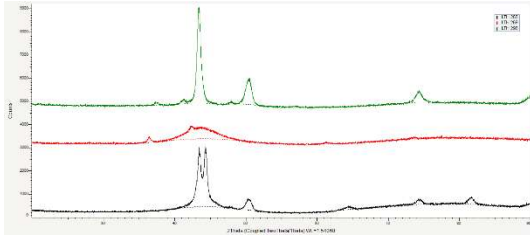


Figure 7. Results of XRD-analysis of solid SLM-samples of three alloys. Green curve = ESA1, red curve = ESA2, and black curve = ESA3.

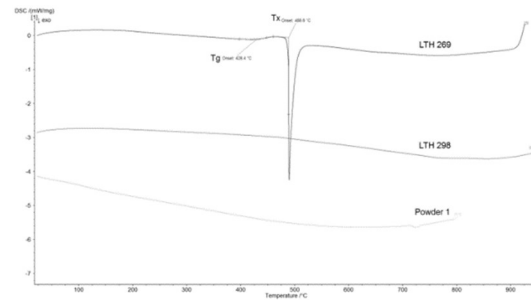


Figure 8. Results of DSC-analysis of alloy ESA1 (solid sample LTH298 and Powder1) and ESA2 (solid sample LTH269). ESA2: $T_g = 428.4^\circ\text{C}$ and $T_x = 488.6^\circ\text{C}$
ESA1: No thermal events

4.3 Washer development (WP3)

Taking all results hitherto into account, it was decided to move forward with alloy ESA1 for the remaining activities. Seven washers with diameter 72 mm were built in this WP.

4.4 Washer evaluation (WP4)

As described in Section 3.4, it had been decided to treat the washers with Hot Isostatic Pressing (HIP) before the Multi Contact Test (MCT) evaluation, in an effort to minimize the number of defects in the material. To define the parameters of the HIP process, the thermal properties of alloy ESA1 had to be measured at this point. Results of the thermal analysis and the subsequent HIP-processing follow.

4.4.1 DSC-results

In Figure 8, DSC-curves for alloy ESA1 (solid sample LTH298 plus Powder1) and ESA2 (solid sample LTH269) are reported. T_g and T_x have been identified for ESA2, as the curve show the typical kinks corresponding to phase transitions. However, the curves for ESA1 (solid and powder) are flat – no events were recorded. This means, unfortunately, that the alloy was more crystalline than previously understood from the XRD-results. Hence, no input for the HIP-processing could be deduced from this analysis.

4.4.2 Results of HIP-processing

Considering the results of the DSC-analysis, it was decided to conduct the HIP-processing nevertheless. While hold temperature and pressure was the same in the two cycles, hold time was set at 10 minutes in HIP-cycle 1 and 2h in HIP-cycle 2. Lacking the input on parameters from the DSC-analysis, hold temperature was selected at the lower end of the T_g interval for Fe-based amorphous alloys, and pressure was set at maximum achievable at that selected temperature.

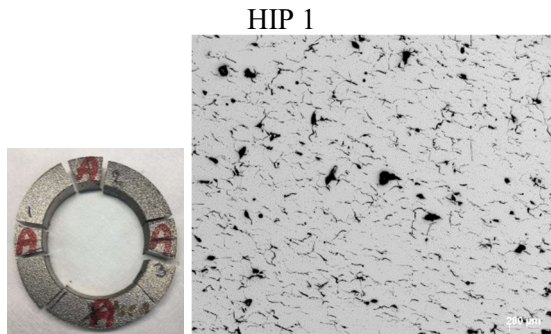


Figure 9. Results of HIP-cycle 1. Scale bar = 200 μm .

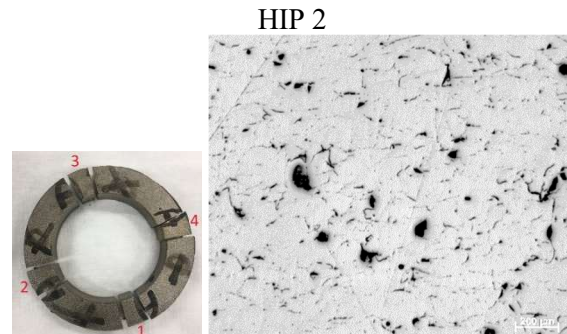


Figure 10. Results of HIP-cycle 2. Scale bar = 200 μm .

Unfortunately, neither HIPped microstructure proved to fulfil the criteria for proceeding with the Multi-Contact test, as stated in Section 3.4.3 of the full version of the Final Report. As the micrographs in Figure 9 and Figure 10 show, no improvement compared to as-built material (Figure 4) is obvious. Sufficient densification had not been achieved, and defects much larger than 1 μm were still noticeable. At this point, no further process iterations were attempted and it was decided to cancel the multi-contact testing.

5 Summary and conclusions

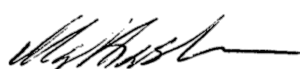
Summarising the outcomes, and comparing them with the technical objectives stated in Section 1.2, it was concluded that most of the objectives were fulfilled:

#	Objective	Outcome
1	To build and evaluate Fe-based AM parts with amorphous microstructure	Yes – Parts of amorphous Fe-based alloys have been built, delivered to partners and to ESA, and evaluated.
2	Part dimension > 1 cm	Yes – Large parts of various dimensions have been delivered and evaluated. Washers were $\text{Ø}72 \times \text{H}12$ mm (1 alloy) Largest cylinders were $\text{Ø}22 \times \text{H}32$ mm (3 alloys)
3	Relative density > 97%	High as-built densities were achieved, but not measured with Archimedes nor any calibrated image analysis tool. Estimated densities, based on previous experience of metallographic examinations of SLM-processed material, are in the 95-98% range.
4	Structure > 80% amorphous	Of three alloys, one was clearly amorphous according to XRD and DSC analyses. Two alloys were amorphous-crystalline composites. The amorphous content was not quantitatively measured.
5	Hardness > 900 HV	Yes – Hardness was measured to 1000 HV for one alloy, and to 830 HV for the other alloy.

During the activity, the challenge of achieving an additional criterion clarified. The threshold for acceptable defect size was set at 1 μm for MCT testing to be worthwhile. Mature alloys in mature processes can fail this criterion. To achieve nearly defect-free material for a completely new alloy in a new SLM-process chain within the scope and timeframe of this activity proved to be too challenging. Therefore, WP4 was re-structured compared to the original plan, and MCT testing was cancelled. Time and resources were instead utilised for the feasibility study of HIP, thereby creating new knowledge for the project partners. In future work, a more comprehensive effort on utilising HIP as a method to improve material quality will be of interest. To ensure that a material with suitable thermal properties for HIPing would be selected in that case, it is understood that DSC analysis should be performed at an earlier stage and results used for evaluation and alloy selection.

The activity has demonstrated the feasibility of processing the identified amorphous alloys with SLM. In addition, valuable insights have been obtained in postprocessing of such parts by utilising HIP to reduce defects in the solid, near-net shape parts. Also, challenges associated with the target application (bearings) have been identified. Work with improving the material quality will continue, but, also, search for potential application of interest where the currently achieved material quality can be used will be a focus in future activities.

Regarding evaluating the technology readiness level (TRL), TRL is still considered to be 2 for the intended bearing application.


Mattias Unosson