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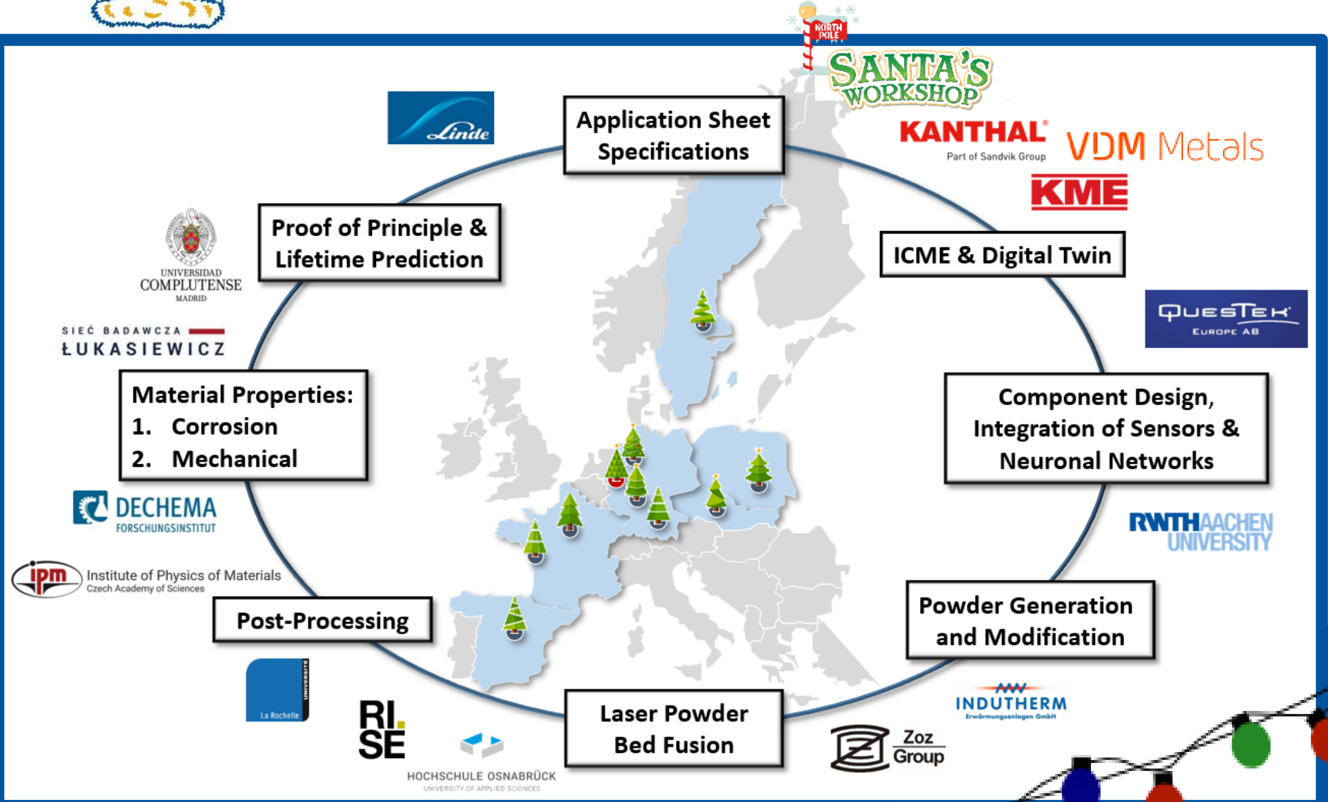
# topAM BRIEFING

*Developing ODS high temperature alloys tailored for AM*




# *Happy Holidays!*

**This Holiday Edition of the topAM Briefing includes the latest project results from modelling the optimal particles, compositions and distributions to the production of samples and testing of the baseline alloys in aggressive environments. Each work package is highlighted in a technical activity summary.**


This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 958192.

## WP2 Optimal computational design for LPBF based on ICME

The effect of dispersed particles on grain structure evolution during rapid solidification has been studied. This is realized via the creation of a two-dimensional multi-phase field model implemented in MICRESS® software which incorporates the seed-density model. The grain structure evolution simulation for a six-component ferritic steel system with the presence of seed particles has been performed and validated by experimental data from literature. The grain evolution simulations enhance the understanding of grain morphology transition from columnar structure to equiaxed structure under different solidification conditions, such that the concurrent design of AM processing conditions and ODS particle distributions can be optimized (Figure 1).

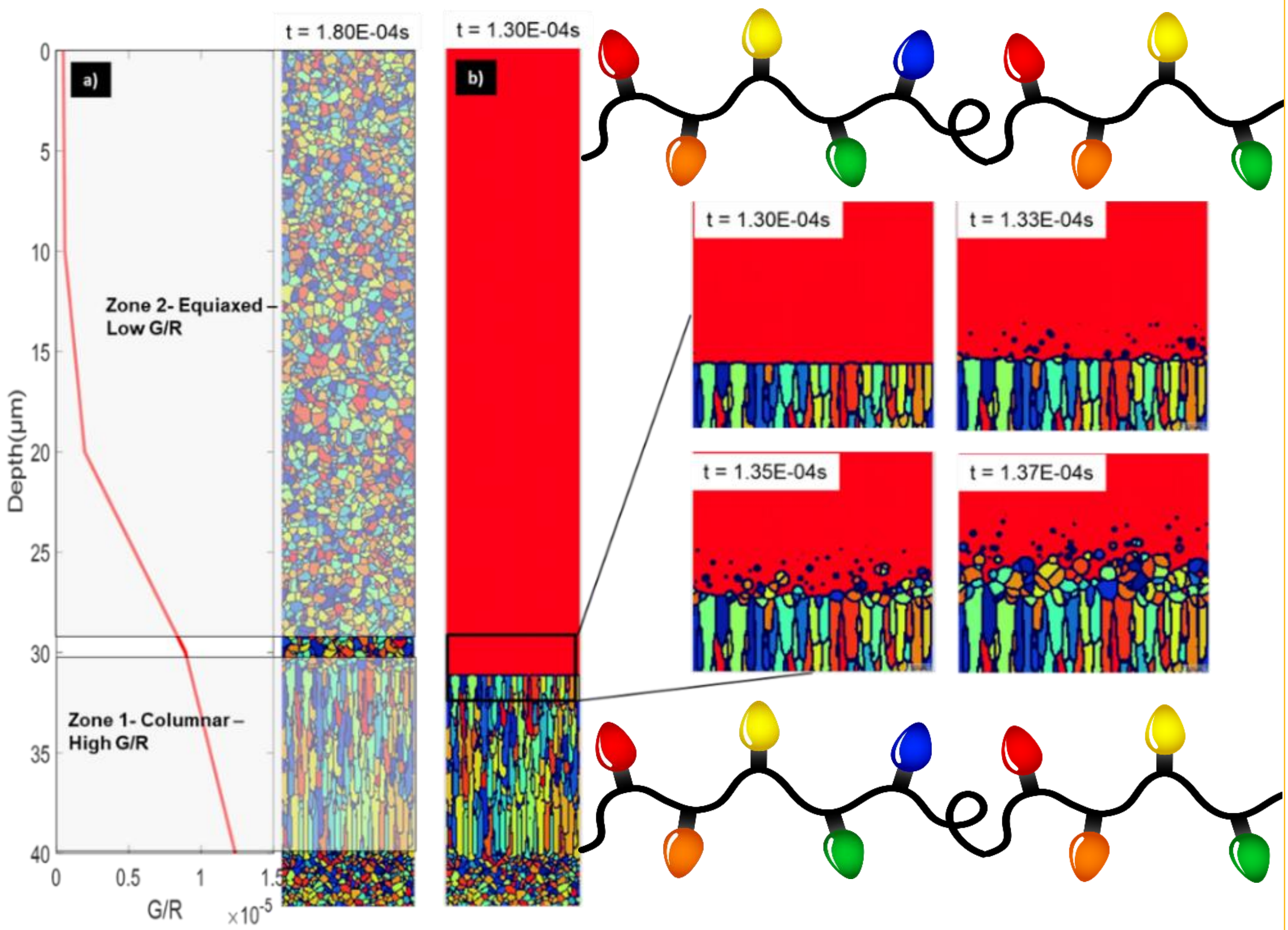



Figure 1. Grain structure simulation indicating the influence of dispersing particles on grain morphology evolution.

## WP3 LPBF component design

Considering the minimum wall thickness, build orientation, and the build volume of the individualized EOS M290 for printing Alloy 400 at FH Osnabrück, the geometries for the heat exchanger components, namely the ferrules and temperature control device, were prepared and delivered to the respective partners.

Additionally, following the successful integration of the Fiber Bragg Grating (FBG) sensors into the channels of a designed and additively manufactured component, an experiment was performed to obtain the temperature data of the component. The component was heated to 1000° C, and the FBG sensor response was plotted against thermocouple measurements. The result of this experiment is shown in Figure 2.

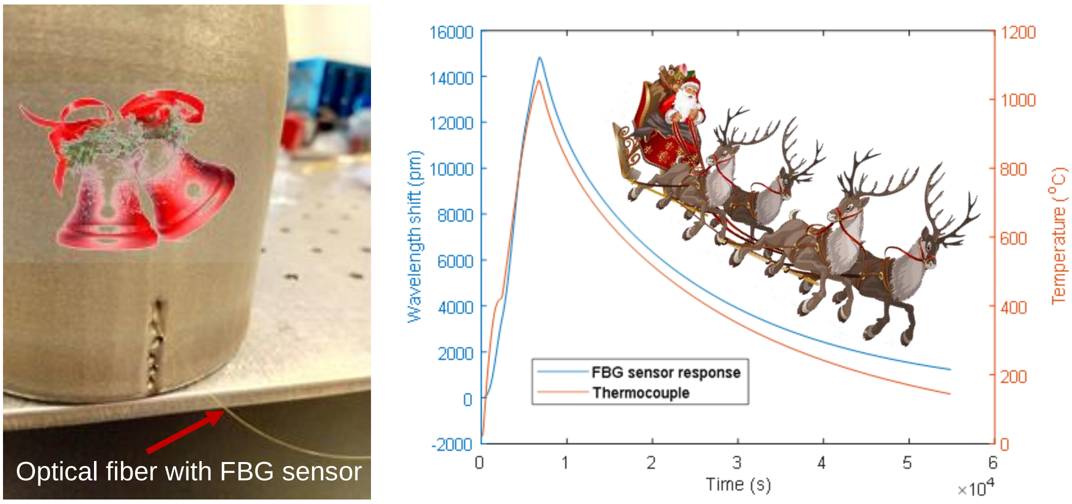



Figure 2. FBG sensor integrated component (left); comparison of the results obtained using the thermocouple and FBG sensors (right).

## WP4 Powder production and modification

After having identified the suitable size and amount (3 vol.%) of yttria-nanoparticles, as well as process parameters, the first batches of freeze-granulated Fe- and Ni-based alloy powders are currently being prepared to obtain sufficient quantities (~ 10 kg) for LPBF printing trials in WP5. As a pre-trial and proof of concept, test cubes using freeze granulated stainless steel powder with 3 vol.% TiC were printed with LPBF, arriving at a printed density of >96% and a homogeneous distribution of nanosized TiC-particles (Figure 3).

While moderately spherical and flowable powder particles eventually have been obtained by freeze-granulation, this remains a challenge for mechanically alloyed powders using high energy ball milling. Figure 4 shows that, starting from spherical as-atomised AM100 powder in the 15-53 µm range and additions of 5 vol.% of nanosized yttria, heavily deformed and flattened particles are obtained after 4 h milling time, which break up into moderately spherical, but very fine and agglomerated particles after 10 h milling time.

For the ‘internal’ oxidation/nitridation process, the dispersoids form between oxygen/nitrogen and specific alloy additions. Suitable amounts of elements (Y, Zr, Ti and Hf) have been identified for the different alloy systems by the simulation work of WP2. Corresponding powders with modified alloy compositions have been produced for post-treatment in a fluidised bed reactor. Next, sufficient quantities for LPBF printing trials will be produced using process parameters formerly identified for unmodified alloy powders.

Batches of powders with modified alloy compositions were also produced using pre-heated nitrogen as an atomisation gas, aiming at nitridation in-situ during atomisation. These have already been provided to WP5 for initial LPBF printing trials. Next steps include atomisation trials using an $Ar\text{-}O_2$ mixed gas to achieve in-situ oxidation. Analysis of powders from pre-trials using the unmodified Fe-based AM100 alloy composition revealed oxygen/nitrogen levels in the 500-1000 ppm range in the as-atomised state. While this is below the targeted values based on the computational alloy design, it is theorized that larger amounts may be absorbed using the modified alloy compositions.

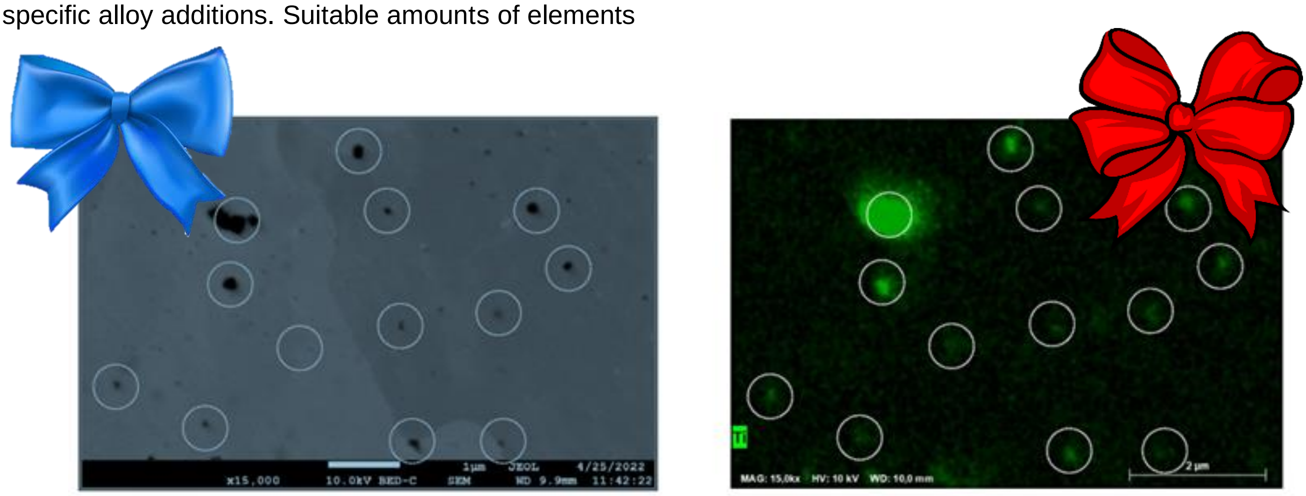



Figure 3. SEM (L) and EDS (R) of TiC particles in LPBF test cubes using freeze-granulated stainless steel powder.

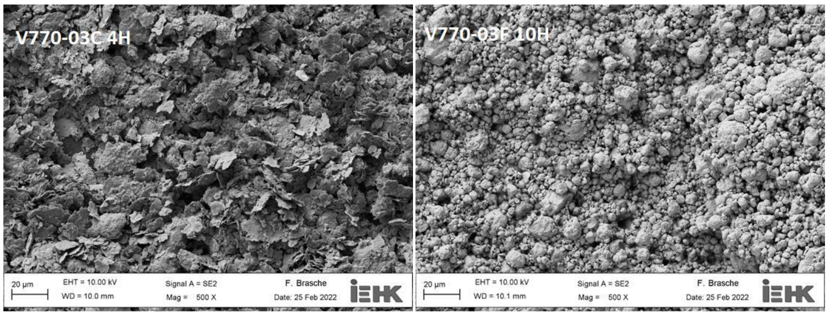



Figure 4: Development of AM100 powder particle shape and size during mechanical alloying (4h left, 10h right) with 5 vol.% nanosized yttria.

## WP5 LPBF and Post-Processing

Post processing investigations have started. Hot isostatic pressing (HIP) has been effectively utilized to further densify the AM produced samples, especially as-printed alloy 400, as seen in Figure 5. In addition, investigations on alloy 699XA and alloy 400 post processing have begun. AM produced alloy 699 XA samples with different surface conditions have been exposed at 620 °C (in an industrial metal dusting atmosphere, Figure 6). Under these conditions a protective chromia scale is formed on the surface of this alloy. The results show that AM processing does not appear to affect the overall metal dusting performance of Alloy 699 XA; however, the ground surfaces appear to form the better protective scale over the as-built, rougher surfaces.

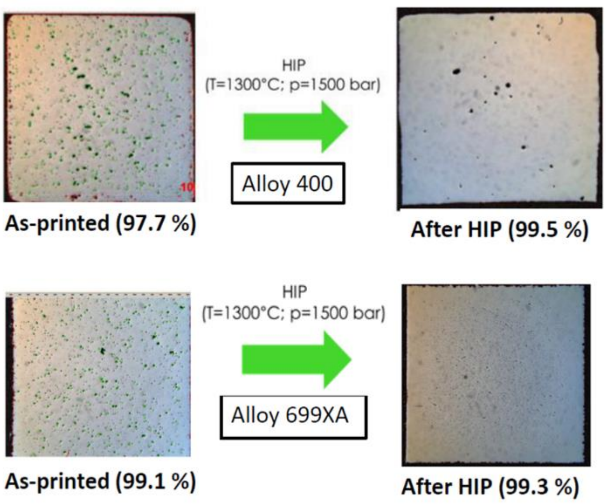



Figure 5. Cross-sections of hot isostatic pressed samples of as-printed Alloy 400 and Alloy 699XA.

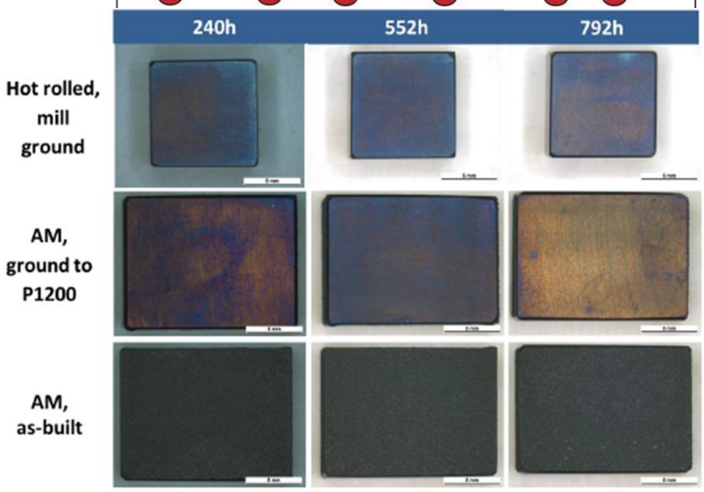



Figure 6. Alloy 699XA AM-produced and hot rolled exposed at 620 °C in an industrial metal dusting environment for different durations with varying surfaces.

## WP6 Material properties

From the initial results obtained during the preliminary and planned screening tests, the Ni-based alloy 699 XA stands out for resisting extreme conditions when compared to the other studied alloys, such us 602 CA and alloy 400. Bulk and cast 699XA samples not only presented an effective response under the different atmospheres but also in terms of mechanical properties. Only under a molten salt atmosphere (60%$NaNO_3$-40%$KNO_3$) did 699XA show slightly worse behavior than 602CA. However, overall performance still indicates that both materials are suitable for long-term testing.

After steam oxidation under different temperatures, densities, and heat treatments, alloy 699XA showed higher oxidation resistance than alloy 400, which suffered severe internal oxidation. Also, preliminary microstructural analysis of 699XA after exposure in air at 1100°C indicated good resistance to oxidation. In the aggressive industrial metal dusting environment of 620°C and 18 bar for ~800h, 699XA presented good behavior (Figure 6 above). Finally, the thermophysical properties and the overall excellent mechanical properties confirmed the excellent material processing via additive manufacturing (Figure 7).

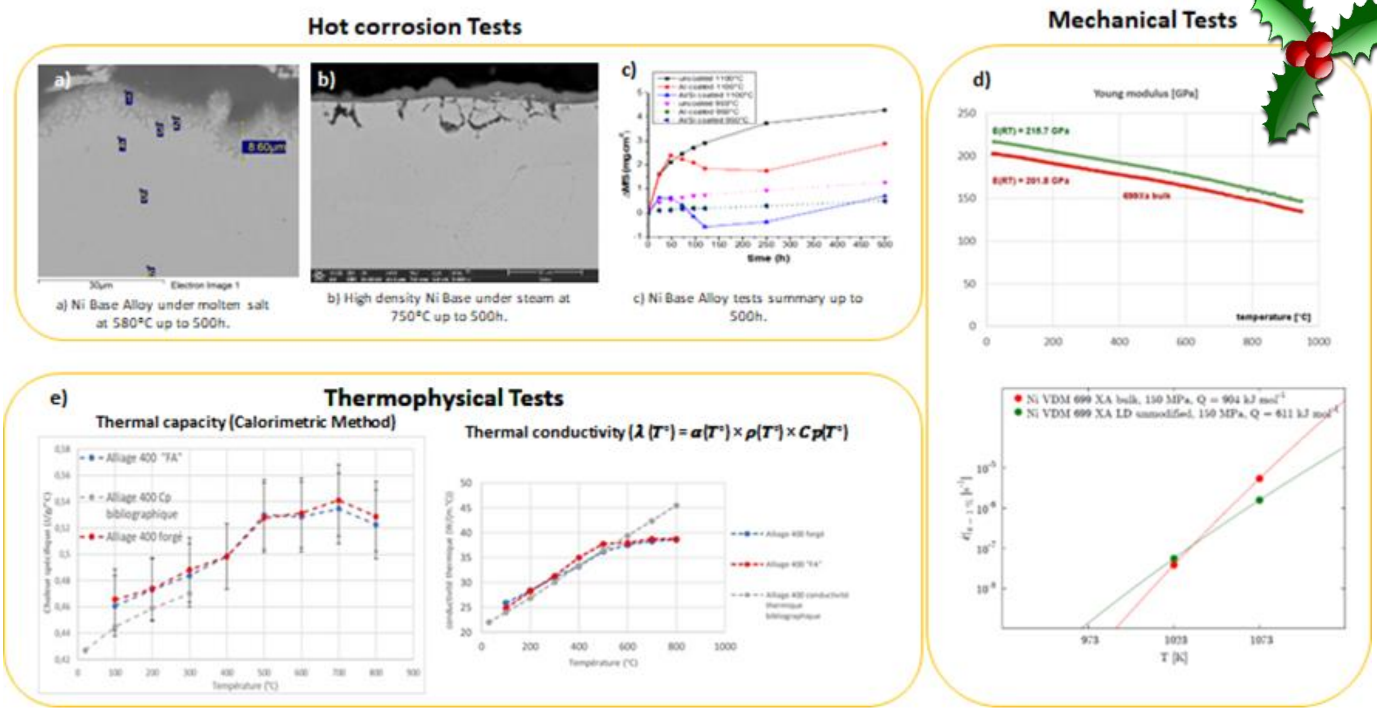



Figure 7. Examples of material property characterization: a), b), c) high temperature corrosion tests; d) mechanical tests; and, e) thermophysical tests.

## WP7 Proof of principle

Planning for the proof of principle validation at the end of the project is already underway, including the LPBF builds and the testing facilities for each condition. These activities are scheduled for later in the project after the major results of the other WPs have been compiled.

Note: the other non-technical project work packages, WP8 Dissemination, communication, exploitation and WP9 Technical coordination and project management, are on target but are not discussed in this briefing.

**How to get more involved:**

**You can follow the latest topAM project activities on LinkedIn (topAM Project) and Twitter (@topAMproject)! These are the best locations to stay up-to-date on the project's communication and dissemination activities. Email [emma.white@dechema.de](mailto:emma.white@dechema.de) to be added to the LinkedIn topAM project group. Also, our project website is regularly updated at [https://www.aspire2050.eu/TopAM](https://www.aspire2050.eu/TopAM).**

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*And Happy New Year!*