

PROCESSING ODS MODIFIED IN625 USING SELECTIVE LASER MELTING

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Abstract

Increasing the operating temperatures of power plant turbine generators is a universal method to increase the efficiency of steam and gas turbines. However, operating a plant at higher temperatures poses extreme challenges to the materials used, especially regarding oxidation, creep, thermal fatigue- and stress-corrosion cracking. The EU-OXIGEN project addresses these issues by the development of novel processing routes for ODS-modified materials, as this class of materials offers exceptionally high temperature strength, oxidation and corrosion resistance at temperatures exceeding 900°C. Additive manufacturing processes such as Selective Laser Melting are considered to enable their successful processing. First results on density of SLM-processed, mechanically alloyed ODS-modified Inconel-625 superalloy powders, are presented and compared to the processing conditions and results of gas atomized conventional Inconel-625 powders. Whereas for IN625 a wide and stable processing window was found, significant differences for the ODS variant in terms of the required laser energy input to reach density values >99% are found. Microstructural analysis of precipitates lead to the conclusion that the milling process for ODS variant is key to achieve good quality materials and results.

Keywords: Selective Laser Melting, Additive manufacturing, ODS materials, Microstructure

Introduction

Additive Manufacturing (AM) Technologies cover a wide range of comparably new production processes, where the parts to be built are manufactured layer by layer [13]. Thereby, the CAD file of a part to be built is sliced into thin layers with a typical thickness between 20µm and 200µm, depending on the specific process and the processing conditions. The raw material is then selectively consolidated within the cross-sections of the part at a specific height. Industrially relevant processes for the production of metallic components include Selective Laser Melting (SLM) and Layer Metal Deposition (LMD), where the energy required to melt a powdered metal material is delivered by a Laser beam. Due to the layer-wise build process, AM processes allow the manufacturing of highly complex shaped parts, which can significantly improve their performance [14] e.g. in terms of lightweight [25], functional [25], and structural optimization and thermal behaviour [10, 19, 23]. As a consequence Additive Manufacturing gains in importance in many industrial fields.

In SLM thin layers of a powdered raw material are deposited, and the energy beam is used to selectively scan the powder surface with the corresponding cross-sections. This leads to full melting of the powder particles exposed to the scanning laser spot and to selective consolidation the powder material, reaching almost 100% material density [24, 33]. SLM

processed materials typically offer properties quite comparable to the properties of conventional bulk materials [15, 28, 32]. There is a wide range of processable standard metal materials available: From stainless and hot-work steel [4, 24], Aluminium [5, 7, 8], Titanium [21] and Ni-based materials [20, 22] are typical metal classes. However, the SLM-process is a master forming process, implying that the final material together with its incorporated properties are generated by the manufacturing process itself. This opens up new opportunities for the development of materials specific applications and sectors. An example are metal-diamond composites, as proposed by Spierings [26], where the diamond particles only can survive due to the very short laser-material interaction time, combined with a very high cooling rate of the melt pool. This basic concept can also be adapted to other classes of materials, such as Oxide Dispersion Strengthened (ODS) materials.

ODS alloys

ODS alloys are an established class of materials that offer exceptional high temperature strength, oxidation and corrosion resistance at high temperatures exceeding 1000°C, along with outstanding resistance to radiation damage. ODS alloys contain transition metal oxides (usually yttrium based), forming fine dispersed particles in the metallic matrix – ideally in the size range of about 5nm to ≈30nm. These particles hinder the movement of dislocations, thereby improving related material properties by the Orowan mechanism.

The history of ODS dates back decades. In the 1960's, Prof. N.G Grant of MIT studied two dispersion strengthening processes involving powder blends of oxides with copper and internal oxidation of copper and nickel alloys, finding dramatically improved high temperature strength. Further research was undertaken by Sadtry, Alexander and Weeks et al. from the Technical Research Associates from NASA. They developed in the '80s techniques for producing castable ODS metals. However, one problem was that the alloys could not be welded nor was the solidification rate sufficiently high enough to maintain a homogeneous particle distribution and to prevent excessive precipitate growth.

Nevertheless, such alloys are envisioned to be used in a number of future fossil energy and nuclear power applications [9], in order to improve their cycle efficiency by increasing the operating temperature of the turbine generator. Today, the inlet and exhaust temperatures of gas turbines are approximately 1500°C and 600°C, respectively, but will increase in the long term with exhaust temperatures exceeding 650°C. Operating a plant at such elevated temperature poses extreme challenges to the materials used; in particular, creep, thermal fatigue cracking and stress corrosion cracking are of major concern. However, while the fundamental material properties are expected to be well suited to turbine applications, the manufacture of components using ODS alloys are currently subject to a number of economic and technical barriers:

- Currently available mechanical alloying processing equipment used to produce ODS alloys are inefficient hence lead to high production cost.
- Oxide particles tend to coarsen when using conventional fusion (high heat input) and joining techniques, which can lead to reduced high temperature creep strength [16-18].
- ODS materials are difficult to repair for reasons given above.
- They are difficult to manufacture with traditional machining techniques (drilling, milling, grinding) due to their superior properties.
- Optimisation of high temperature creep strength in an ODS material requires recrystallization which produces coarse, usually high anisotropic grain structure.
- Coarse grained ODS alloys can give significant component to component variability in creep life. Moreover, these alloys tend to be creep brittle (e.g. <1% creep elongation to failure), so there can be little warning of imminent failure using time averaging approaches, increasing the risk of unplanned downtime.

Using the SLM process therefore opens up an alternative way towards manufacturing structurally optimised components out of ODS alloys, as the process-specific advantages such as short melt-pool lifetime and high cooling rate will favour superior ODS material properties by maintaining fine dispersed and homogeneous ODS particle distribution in the alloy matrix. The basic concept was already investigated by Walker [31] and Boegelein [6] for the Fe-based ODS alloy PM-2000 manufactured by Plansee. Boegelein concluded that a “*fine dispersion of precipitates was retained in the SLM builds, and findings suggest that a certain amount of Y is probably still in atomic solution in the as-grown condition and forms new small nanoscopic dispersoids during annealing, which lead to enhanced strengthening*”.

The EU-OXIGEN project aims at the development of ODS modified super alloy powders and manufacturing routes for turbine applications. The current paper presents first results on SLM-processed conventional IN625 and a corresponding ODS modified alloy variant. The preliminary results show that there are many challenges to be addressed in order to reach high-density SLM parts.

Materials and Methods

Materials

In order to build a comparison basis, a standard IN625 powder material with a particle size distribution suitable for additive manufacturing was used. The alloy composition is shown in Table 1.

Table 1: Measured alloy composition of IN-625 powder

Element	Ni	Cr	Mo	Nb	Fe	Si	C	O	other
wt (%)	bal.	20.83	9.10	3.64	3.60	0.10	0.02	0.013	0.17

This powder raw material was also used to process an Yttrium oxide (ODS-) alloy variant, using High Energy Ball Milling (HEBM) techniques. Therewith, a material composition of 99 ± 1 wt% of IN625 and 0–2 wt% of nano-yttrium oxide was achieved, named IN625-ODS.

HEBM processing

A High Energy Ball Milling (HEBM) processing route, as described by Suryanarayana [30], has been specifically developed by MBN using a proprietary production plant for the manufacture of IN625-ODS powder for additive manufacturing. The challenge of milling IN625 base materials is closely related to the tendency of the powder particles of cold welding together during the process. This phenomenon might be prevented by an efficient dispersion of Yttria nanoparticles in the IN625 matrix, and modifying the HEBM processing parameters allows the balancing of the impact energy and therefore the fracturing behaviour and welding mechanisms of the particles. This results in improved dispersion homogeneity, and process yield of particle fractions suitable for SLM (Figure 1). Powder post-processing can be applied to increase this yield for suitable particle morphologies. HEBM processing and post-processing was performed in an inert atmosphere, minimizing the uptake of contaminants – mainly Oxygen and Nitrogen. Never the less, it was observed that by HEBM the O-content typically was increased to values in the range of $\approx 0.7 - 1.0\%$, and the N-content up to $\approx 0.25\%$.

SLM Machine and processing parameters

The materials have been processed by SLM using a ConceptLaser machine type M2. The machine is equipped with a 200W fibre laser with measured wavelength of 1071nm, operated in cw-mode. The scan strategy used to produce the $10 \times 10 \times 10 \text{ mm}^3$ (IN625) and the $8 \times 8 \times 3 \text{ mm}^3$ (IN625-ODS) test samples, respectively, is a meander-like scan strategy where the scan

direction is turned by 90° for each layer, corresponding to the standard scan strategy used on ConceptLaser machines.

Table 2: A range of SLM processing parameter were applied for IN625 and IN625-ODS

	Layer thickness t (μm)	Scan speed v_s (mm/s) *	Hatch distance d (μm) *	Energy level E_L (J/mm^3)
IN625 / IN625-ODS	30	$v_1 - v_2$	$d_1 - d_2$	$E_1 - E_3 \approx 3.5 \times E_1$

*No specific processing parameters are shown here.

Powder particle size distribution

The PowderShape™ system analyses optically several thousand single powder particles (typically $> 10^4$) with an appropriate filtering mask, making sure that single particles are analyzed and no agglomerates of particles. According to Dvorak [11] the method delivers comparable results as laser scattering methods. For atomized powders, such as IN625, the filtering mask selects particles with a diameter between $4\mu\text{m}$ and $75\mu\text{m}$. For milled powders, the filtering range was increased to $120\mu\text{m}$ as such particles are typically somewhat bigger, and more irregular in their shape. A particle diameter is considered as the diameter of a coextensive circle diameter.

Measurement of mechanical properties

Mechanical material properties are measured on horizontally and vertically built and post-processed (drilled) test samples in the heat treated condition, according to EN-10002/ ISO-6892. The applied heat treatment follows the recommendation of AMS-2774 (1038°C for 1h) [1]. A Walter&Bai tensile testing machine type LFV-25, equipped with clip gauges type MFA 25 / 12 is used. The sample diameter in the gauge section was 5mm.

Sample Characterization

The SLM processed samples were analysed with regard to their material density using optical microscopy on polished samples and the Archimedes methods, as described in more detail by Spierings [27]. The microstructures of polished and etched (Kallings no.2) specimens were analysed using both optical and electron microscopy techniques. Furthermore, transmission electron microscopy (TEM) technologies were used to characterize IN625-ODS samples to get insight into material microstructure, and especially into ODS particles and precipitates.

Thin lamellae TEM specimens were produced in a dual beam FEI Helios 600i Focussed Ion Beam (FIB) instrument using the lift out method [12]. TEM analysis was performed using a 200kV JEOL 2100FCS microscope fitted with a windowless EDAX energy dispersive x-ray detector.

Results and Discussion

Powder raw material

The particle size distributions of the gas atomized IN625 is significantly different to the HEBM-processed IN625-ODS powder. The IN625 powder particles are distributed with a median value of $24\mu\text{m}$, whereas the median of the ODS powder particles is about $17\mu\text{m}$. However, the corresponding volume-based D_{10} , D_{50} , and D_{90} values were comparable, with $22\mu\text{m}$, $35\mu\text{m}$ and $51\mu\text{m}$ for IN625 and $22\mu\text{m}$, $41\mu\text{m}$ and $58\mu\text{m}$ for IN625-ODS, respectively. The particle size distribution of IN625-ODS powders could be described by a Log-Normal distribution, whereas the IN625 powder shows more a bi-modal distribution with a small amount of fine particles in the range up to $\approx 15\mu\text{m}$, as shown in Figure 1.

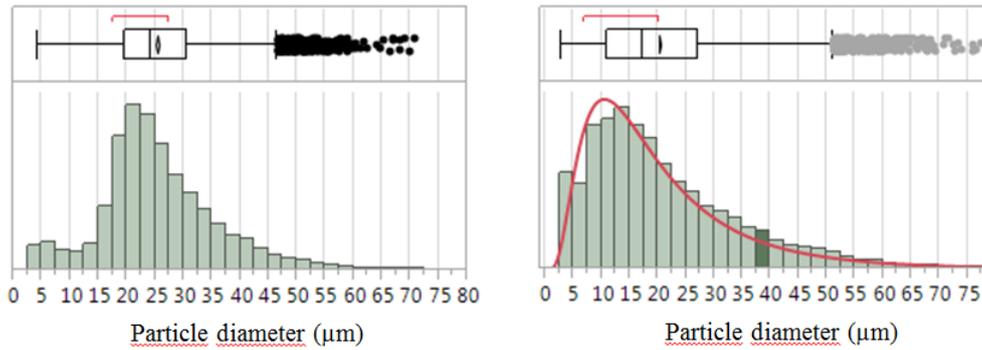


Figure 1: Particle size distribution of the IN625- (left) and IN625-ODS (right) powder. Furthermore, the powder can be distinguished in more detail using the Ellipticity E as the ratio of the major to the minor axis of a coextensive ellipse for each particle. The IN625 particles are more spherical with $E = 1.25 \pm 0.26$, whereas the IN625-ODS particles are more elliptical ($E = 1.50 \pm 0.37$) aligned with a higher shape variation and a more irregular particle surface, as shown in Figure 2 (middle and right).

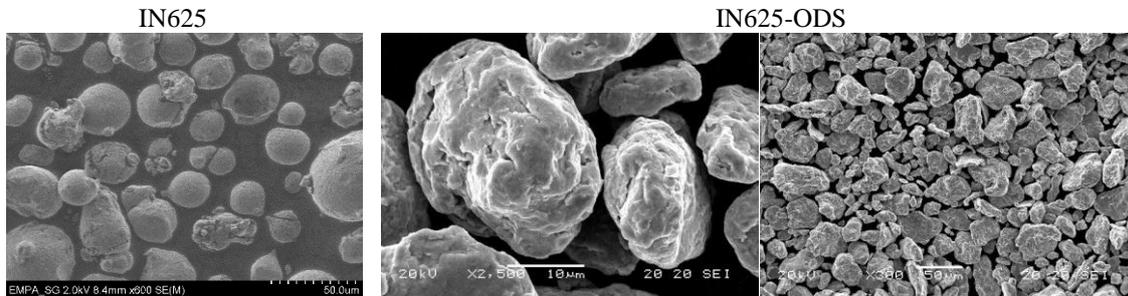


Figure 2: Powder particles for IN625- (left) and IN625-ODS powder (middle and right)

Both powders displayed good flowability with regard to the requirements for SLM, as described in Spierings et.al [29].

SLM processed material density

Figure 3 shows the material density reached after SLM-processing of the powders according to the processing parameter ranges given in Table 2. It was clear that the powders – although similar in composition – behaved significantly different. As expected, the gas atomized IN625 powder reaches a very good material density of $> 99.5\%$ after having reached a certain critical volume energy E_L , which is calculated as $E_L = \frac{P}{v_s \cdot t \cdot d}$ (1)

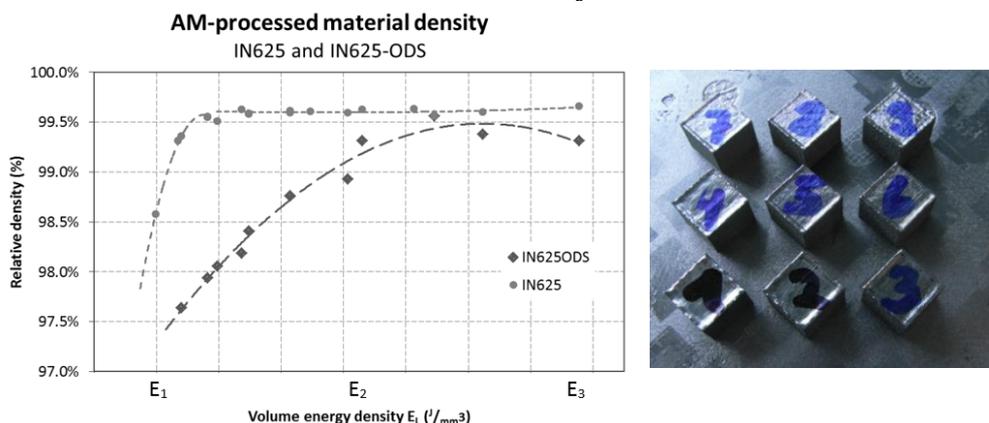


Figure 3: Left: Material density of SLM-processed IN625 and IN625-ODS over volume energy density between E_1 and E_3 ; SLM IN625-ODS samples built on a steel base plate (right).

In contrast, the ODS variant does not show a stable and wide processing window. A comparable density value can only be reached for a significant higher energy input of E_L , which is roughly double the value for the atomized IN625. Micro-graphical analysis show an increasing micro-porosity at lower E_L -levels (Figure 4). This correlates well with density behaviour as shown in Figure 3. In contrast, the IN625 microstructure is very homogeneous, without any cracks or bigger pores inside the consolidated material (Figure 4 right), reflecting the stable and wide processing window shown in Figure 3.

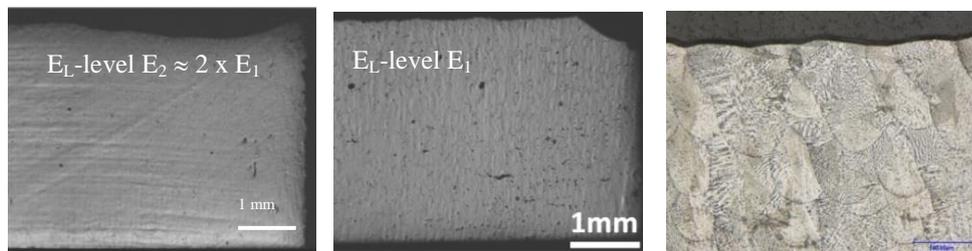


Figure 4: Micrographs for ODS-samples (left, middle) and etched IN625 sample (right)

Reasons for these differences are expected to be the fact that the powder layer density of milled powders is significantly lower ($\approx 30\%$) compared to the layer density of the atomized IN625 powder ($\approx 45\%$), which is due to the irregular particle shape and surface. In addition, the resulting microstructure with oxide particles / precipitates is leading to a different material morphology, where even the precipitates are expected to be porous.

Mechanical properties of SLM-IN625

In order to build a comparison basis, heat treated SLM-processed IN625 material was tested at room temperature so far for its static mechanical properties. Table 3 gives an overview on current mechanical properties.

Table 3: Static mechanical properties of heat treated SLM-IN625 at room temperature

Orientation	Vertical	Horizontal	Anisotropy
Yield Strength (MPa)	579 ± 5	734 ± 3	-21 %
Ultimate Tensile Strength (MPa)	888 ± 6	1036 ± 3	-14%
Young's Modulus (GPa)	159 ± 5	200 ± 3	-20%
Elongation at break (%)	40 ± 1	36 ± 0.3	+11%

The very low standard deviation of 5 samples per orientation reflects the good processing window, and the comparably low level of defects. The measured properties outperform the requirements given by ASTM-3056 [3] significantly, and reach almost the requirements given in the ASM Handbook [2], where the required Ultimate Strength is just the mean value of the measured properties. However, an exception is the Elongation at break where 50% would be required. This indicates a somewhat higher brittleness compared to conventional cast material.

Microstructural analysis

For the ODS-modified IN625 material, all samples in an Energy level range E_L between E_1 and $2 \times E_1$ show a similar distribution of Nb-Cr-Y oxides in the size range of 30nm to about 500nm, as shown in Figure 5 (left pictures). This is somewhat in contrast to findings of Boegelein [6] for Fe-based PM2000, where a comparably fine dispersion of nano-sized precipitates was found after annealing. It is expected that this was due to the retention of Y in solid solution in the as-built condition as a result of the very high cooling rate, which prevented formation and growth of precipitates.

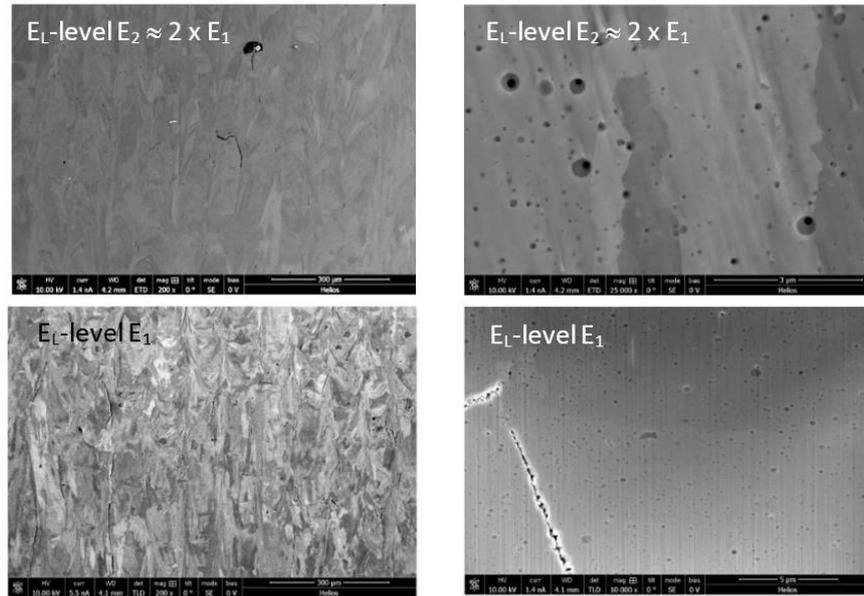


Figure 5: Microstructural analysis of IN625-ODS. Bottom: Energy level E_1 , Top: Energy level $E_2 \approx 2 \times E_1$

In addition, in all manufactured samples grain boundary cracks in parallel to the build orientation (z-direction) are visible, with an indication of increasing crack length with increasing scan velocity (and a corresponding higher cooling rate). This might also be affected by a weakening effect of the coarse precipitates, in combination with internal stresses in the material, as in pure IN625 such cracks could not be found.

The analysis of Nb-Cr-Y-Al oxide precipitates in the IN625-ODS material shows a complex nature of these particles (Figure 6).

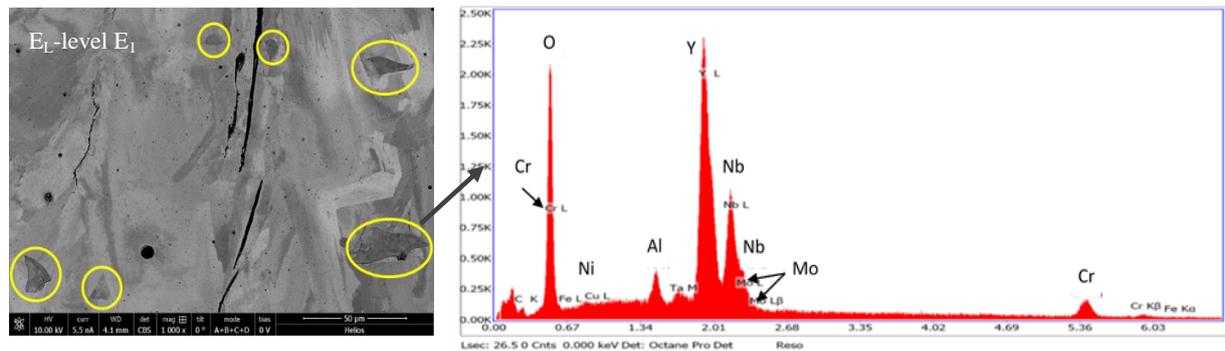


Figure 6: Top: Microstructural analysis of precipitates in IN625-ODS ($E_L = E_1$), showing mainly signals of Nb, Cr, Y, Al, and O.

The precipitates contained some internal porosity, which might have contributed to the overall porosity of the IN625-ODS material, at least to some degree. The precipitates, in general, were polycrystalline, with unidentified crystal structure, as the fast Fourier transforms (FFT) of the TEM lattice images (Figure 7) were ambiguous.

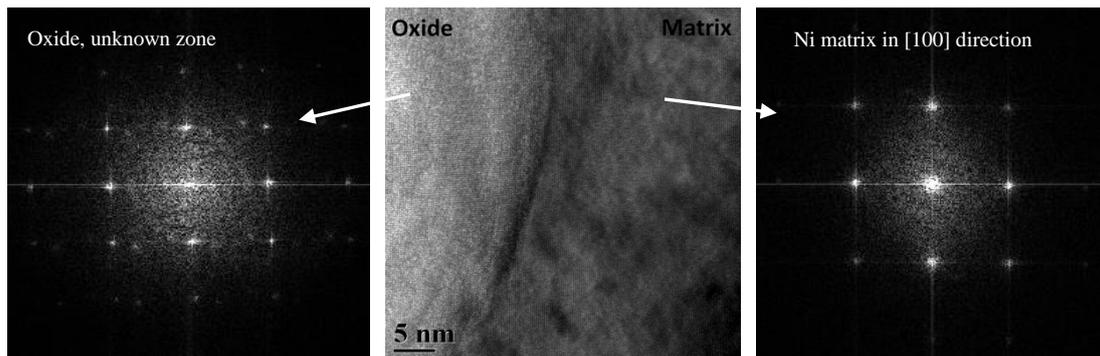


Figure 7: Microstructural analysis precipitates in IN625-ODS. $E_L = E_1$.

Conclusions

The results so far indicate that it is possible to additively manufacture structures from mechanically alloyed powders with a more irregular particle shape. However, the processing window can be influenced significantly, as a higher energy input can be required to reach full material density. In any case, the quality of the processed IN625-ODS material does not yet fulfil the requirements in terms of a stable and wide processing window, and in terms of the resulting material micro structure. Main limitations are the material porosity, ODS particle size and distribution, and the evolution of cracks.

Several aspects have to be considered therefore, aiming principally on a SLM-processing window where an as low energy input as possible is required. Achieving this would better minimize precipitation formation and growth. A supplementary strategy could be the definition of a SLM processing window, where the material density reaches values in the range of about 97%, with a corresponding lower laser scan velocity. An additional HIP procedure could then be applied in order to increase material density, and to start formation of homogeneously distributed of fine precipitates.

This can be achieved by optimizing the particle size distribution, and particle morphology. It is expected that the very wide processing window for IN625 is also a result of an optimal particle size distribution, resulting in a higher powder layer density compared to IN625-ODS. Furthermore, the processing route of mechanical alloying of IN625 with Y should be investigated, thereby minimizing uptake of O and N.

Outlook

Further SLM-processing trials with other, improved IN625-ODS powders will follow. Appropriate microstructural investigations are needed to characterize in more detail the formation of precipitates. Finally, the mechanical material properties have to be measured, in combination with material oxidation resistance. These results have to be compared to the behaviour of conventional IN625, where comparably good properties can be achieved using Selective Laser Melting.

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