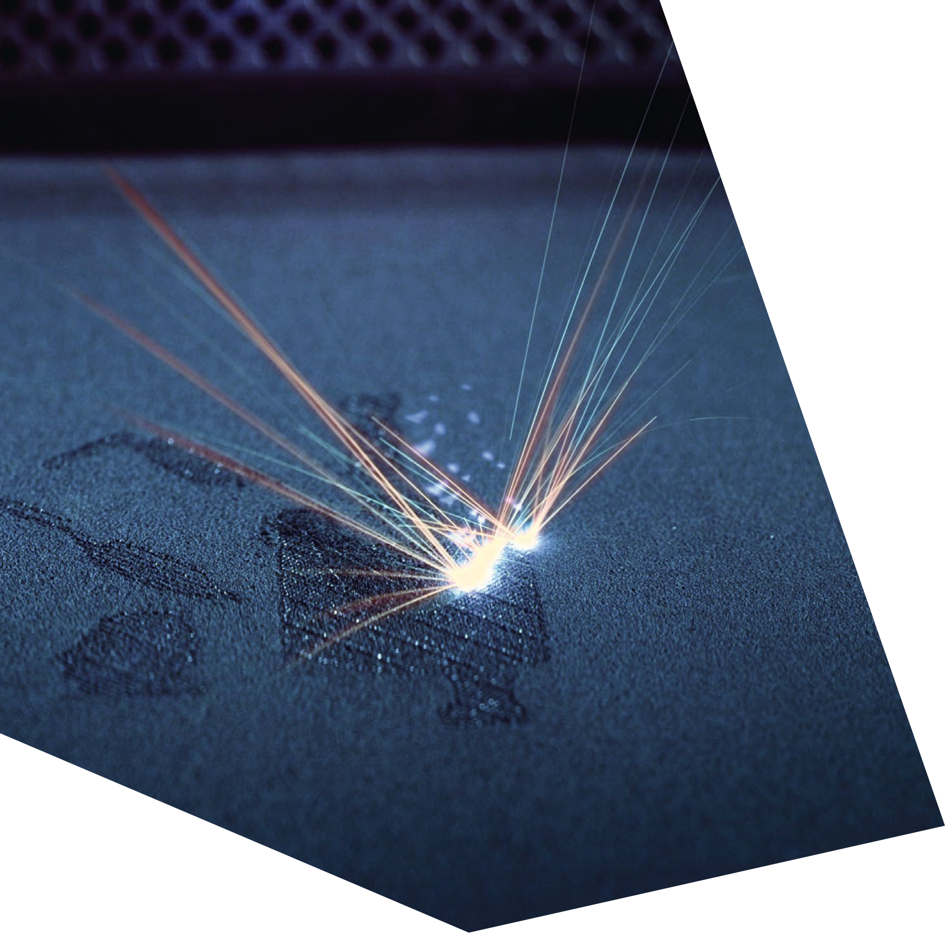

Whitepaper

This whitepaper provides insight into:

- → Processing of AlSi10Mg
- → Atmospheric monitoring and optimization with Linde's ADDvance® O_2 precision
- → Use of EOSTATE Exposure OT with argon-helium process gas

The Influence of Process Gas on Additively Manufactured AlSi10Mg

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How residual oxygen influences the processing of AlSi10Mg and its material quality

EOS and Linde combined their expertise to jointly undertake research in the field of additive manufacturing and material-gas interaction. The aim of this study was to investigate the influence of the oxygen concentration on the properties of AlSi10Mg. The influence of impurities in the process gas on the quality of the additively manufactured material was analyzed, as well as the influence on the process itself.

The results presented in this paper demonstrate which oxygen levels are acceptable for the processing of AlSi10Mg and show how the equipment can be further optimized for specific applications.

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Introduction

In metal laser powder bed fusion with DMLS® technology, inert gases are used to shield the melt pool and avoid impurities and adverse reactions within ambient atmospheres. This also makes the process safer and delivers greater reproducibility and part quality.

The DMLS printing process usually takes place under an argon or nitrogen atmosphere, which is established by purging the build chamber with high purity gas, then replacing the relative ratio of ambient air until an oxygen concentration of less than 1 000 ppm is reached.

Under certain conditions, even after the most rigorous purging of the atmosphere, minor impurities may still remain, as shown in Figure 1. Even extremely small variations in oxygen levels can impact the mechanical properties of alloys sensitive to oxygen, including process-induced aging of the metal powder. As part of the investigations into the material-gas interaction, ADDvance® O_2 precision from Linde was used to provide continuous analysis of the gas atmosphere. If an oxygen concentration as low as 10 ppm is recognized, the unit initiates a purging process to maintain optimal atmospheric conditions. Control and regulation of oxygen is necessary for both quality of the build and reproducibility.

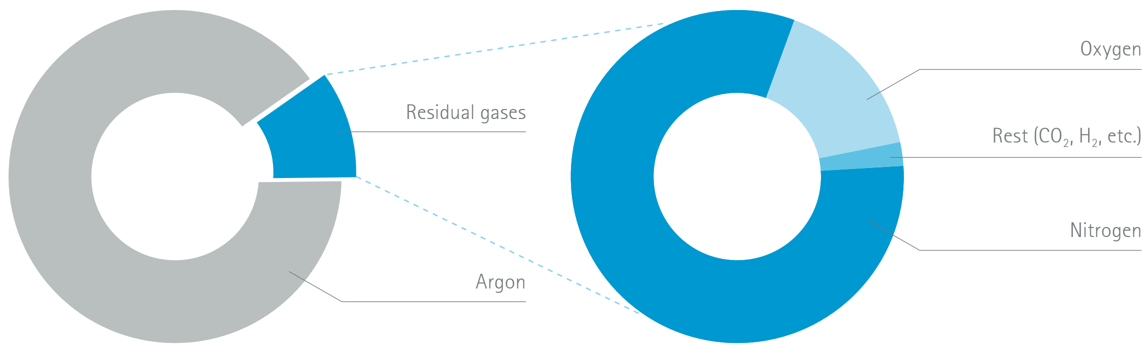

Figure 1: Composition of the atmosphere in the build chamber

Experimental Setup

The material used for this investigation was AlSi10Mg - a widely used aluminum alloy in several industries. The investigations were undertaken using an EOS M 290 system with a 400 W laser, a platform temperature of 35 °C and EOS Aluminium AlSi10Mg powder. Several build jobs were performed with varying oxygen concentrations within the process gas atmosphere. To compare these to the residual oxygen level, three oxygen sensors were used: one at the top of the process chamber, one in the recirculating filter system, and the other in the ADDvance O_2 precision system. This device was carefully calibrated to a level of 500 ppm oxygen[1] to guarantee a precise measurement. The sensors for the control mode within the device show no cross-sensitivity to other gases. The EOS M 290 measures the oxygen level on the top right corner of the building chamber, as opposed to the ADDvance O_2 precision, which measures the oxygen level inside the suction nozzle, close to the build platform.

Gas management system operational scheme

1 Purging gas for sensor protection
2 ADDvance O_2 precision system
3 Sample gas to EOS M 290
4 Sample gas from EOS M 290
5 Inert gas flow controlled by ADDvance to EOS M 290
6 Inert gas supply to ADDvance
7 Inert gas supply to EOS M 290

The tensile specimens were machined in the "as manufactured" state, according to DIN 50125 Type A and tested according to DIN EN ISO 6892-01. All relative porosity values were measured by optical microscopy.

Experimental setup of gas management. (Source: Linde)

To evaluate the influence of the process gas on the material properties of AlSi10Mg as a function of the residual oxygen, 21 build jobs were produced on the EOS M 290 system, using EOS' AlSi10Mg 30 µm process parameters. The only difference between these jobs was the level of residual oxygen.

The jobs were set up with seven different oxygen levels (three per build) to enable a statistical evaluation. The adjusted oxygen levels can be seen in Table 1. To establish a benchmark, the first three build jobs were undertaken using standard conditions. For these, the ADDvance O_2 precision was set to analyzing mode only and tracked the oxygen level. For the other jobs, ADDvance O_2 precision was set to control mode and took over the regulation of the oxygen level within the process gas.

Build Job No.	Oxygen level [ppm]	Linde ADDvance O_2 precision mode
1 – 3	Standard (< 1 000) - benchmark	Analyze
4 – 6	1 000	Control
7 – 9	500	Control
10 – 12	300	Control
13 –15	100	Control
16 – 18	30	Control
19 – 21	5 000	Control

Table 1: Overview of the build jobs, oxygen level and sensor mode

[1] Calibration gas 500 ppm oxygen and balanced argon

The job layout shown in Figure 2 was used to investigate the mechanical properties and porosity.

For fatigue tests, a layout with 15 horizontal cylinders was built three times with a standard oxygen level of < 1 000 ppm, controlled to 1 000 ppm and 100 ppm residual oxygen. The cylinders were tested (R = 0.1) and machined (Type A, D=12.5 mm) analogously to the DIN6072 standard without any heat treatment.

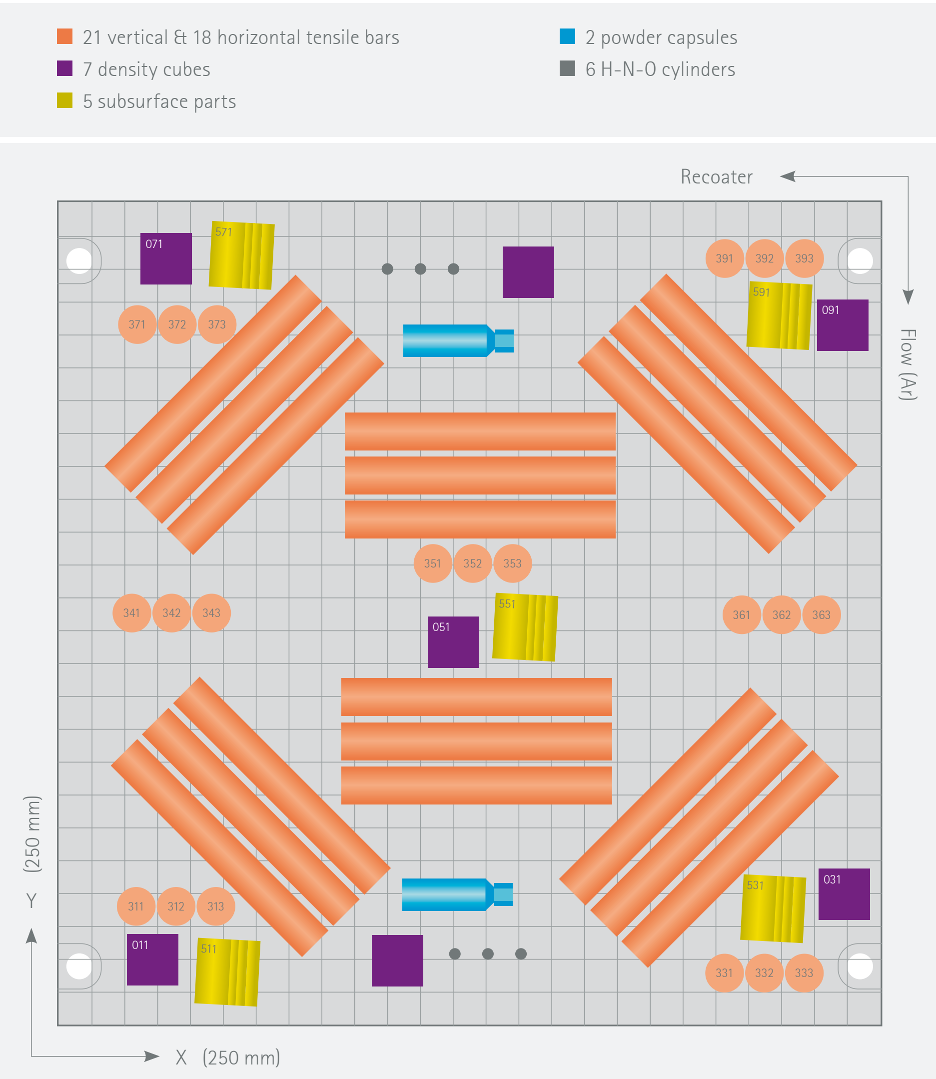

Figure 2: Job layout

Results & Discussion

Oxygen level

The EOS M 290 process chamber oxygen sensor used in the benchmark with the EOS standard atmosphere (oxygen level ≤ 1 000 ppm) shows an apparent constant level of 1 000 ppm. By comparison, the exact values measured by the calibrated ADDvance O_2 precision system are even lower, as shown in Figure 3a. After several layers, the oxygen level measured by the ADDvance O_2 precision quickly drops to around 450 ppm, where the level remains almost constant until the halfway point of the build, when it starts to decrease further.

While the ADDvance O_2 precision system maintains the oxygen level at exactly 100 ppm, a different trend can be seen in Figure 3b. At the beginning of the build, both sensors show almost the same value. However, after some build layers, the signal from the EOS M 290 process chamber sensor rises to over double that of the controlled value, and it continues to rise.

Trends such as those shown in Figures 3a and 3b led us to assume that sensor position is essential for a precise and reproducible measurement. The oxygen sensors in the recirculation filter system of the EOS M 290 (in purple) and the ADDvance O_2 precision system (in blue) follow a similar trend/curve to the signal from the oxygen sensor within the build chamber of the EOS M 290 (in red), with an offset. The results also show that it is possible to precisely monitor and control the residual oxygen within the process gas – in a reproducible manner – to the low value of 100 ppm.

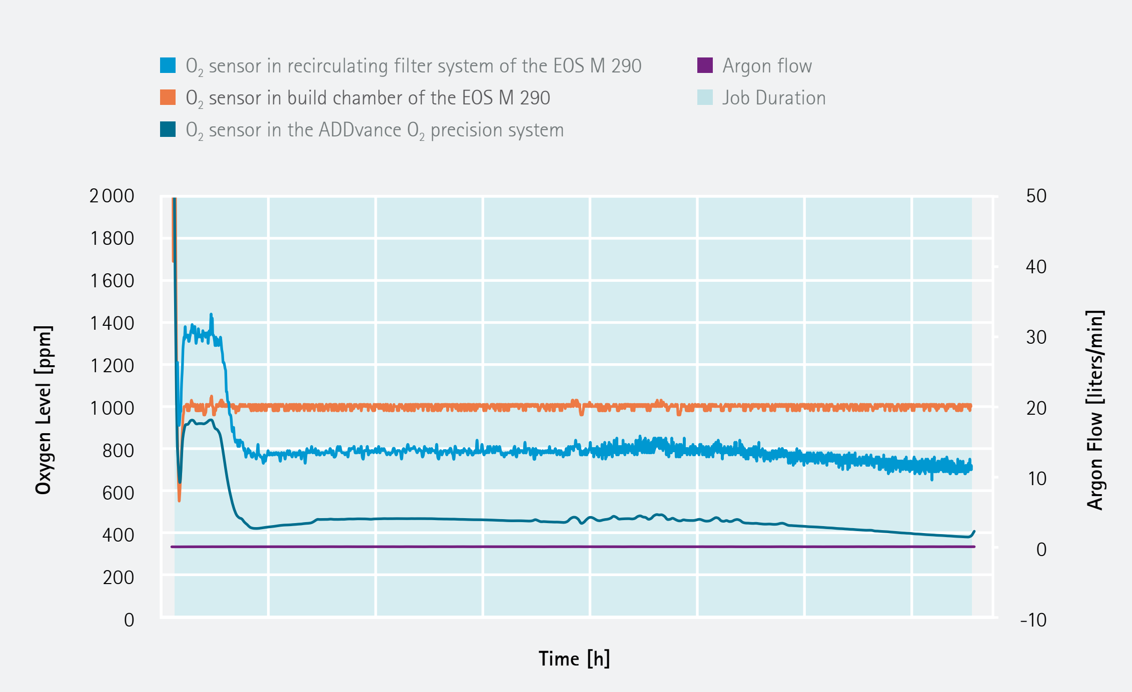

Figure 3a: Oxygen level of build job < 1 000 ppm (EOS standard)

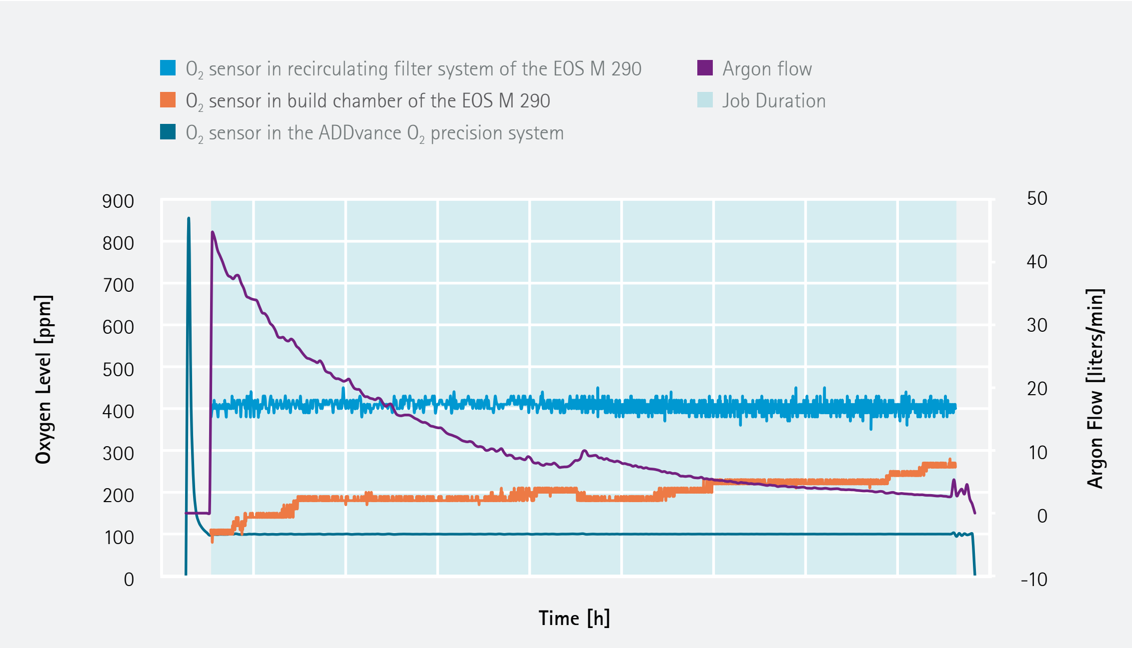

Figure 3b: Oxygen level of build job 100 ppm

Porosity

The colored circles in Figure 4 show a correlation between the level of residual oxygen, porosity and the maximum defect size. It can be observed that, at higher oxygen levels, the relative porosity is also higher, and the size of the pores increases. With lower oxygen content, the deviation of the porosity and maximum defect size is smaller. The 5 000 ppm build job shows a slightly higher deviation in relative porosity. One reason could be the high oxygen level, which leads to discontinuities in the process.

The results confirm that an oxygen level below 1 000 ppm is necessary for consistent material density.

SEM pictures of the used powder and spatter processed under 5 000 ppm oxygen show a change in the morphology and surface chemistry, as seen in Figure 5. This change is probably induced by the formation of oxides or hydroxides, due to a high amount of residual oxygen within the process gas.

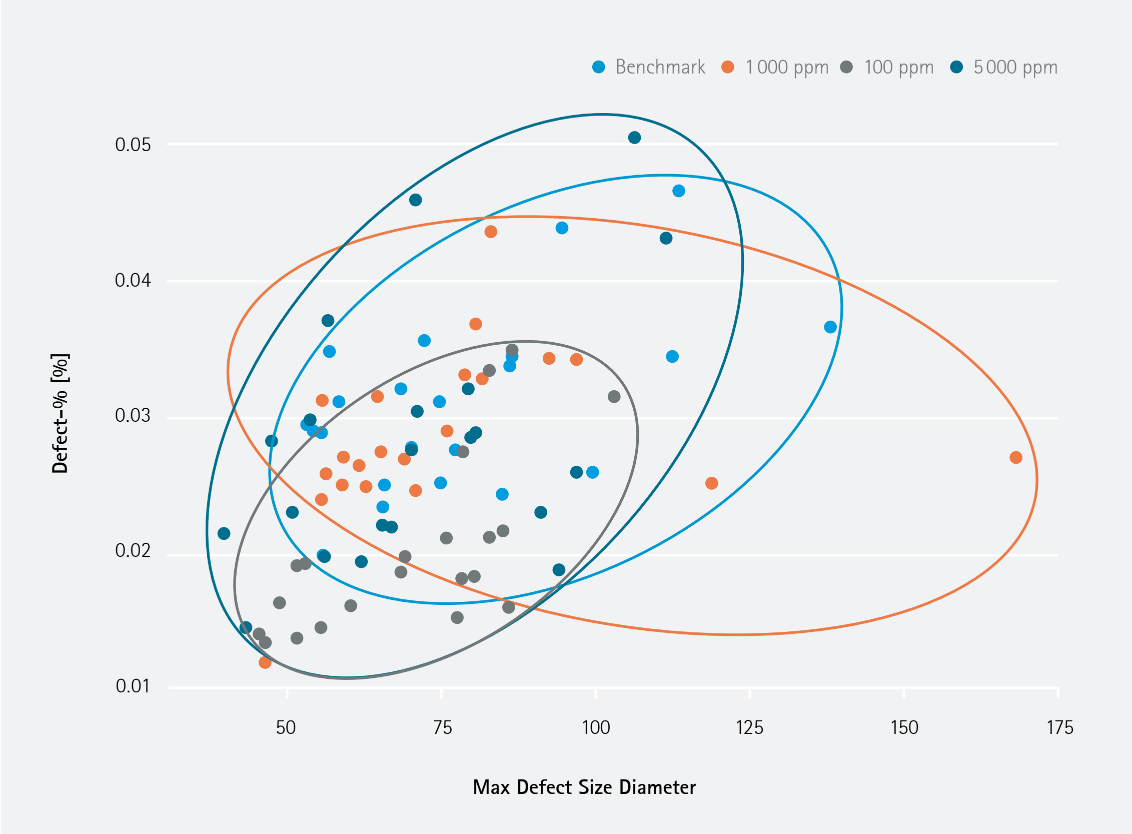

Figure 4: Defect percentage and maximum defect size depending of the oxygen level. Each dot represents one density cube.

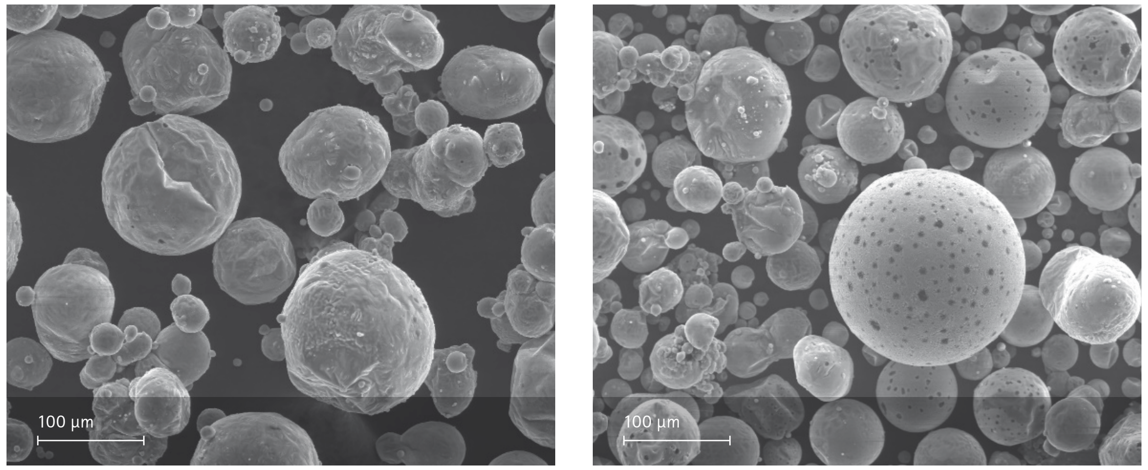

Figure 5: SEM images (500 X) of powder particles collected in the suction nozzles after the build for the EOS standard atmosphere condition (left) and for a process gas atmosphere with 5 000 ppm of residual oxygen (right).

Tensile Testing

The measured values shown in Figure 6 for the Ultimate Tensile Strength (UTS) reached 480 MPa in the vertical direction and 460 MPa in a horizontal direction for the displayed oxygen levels. In addition, Yield Strength values of 230 MPa and 270 MPa were measured, as well as Elongation values of > 8 %.

The results of the mechanical properties stayed at almost the same level as the EOS datasheet[2], which confirms a stable AlSi10Mg process for the EOS M 290 system.

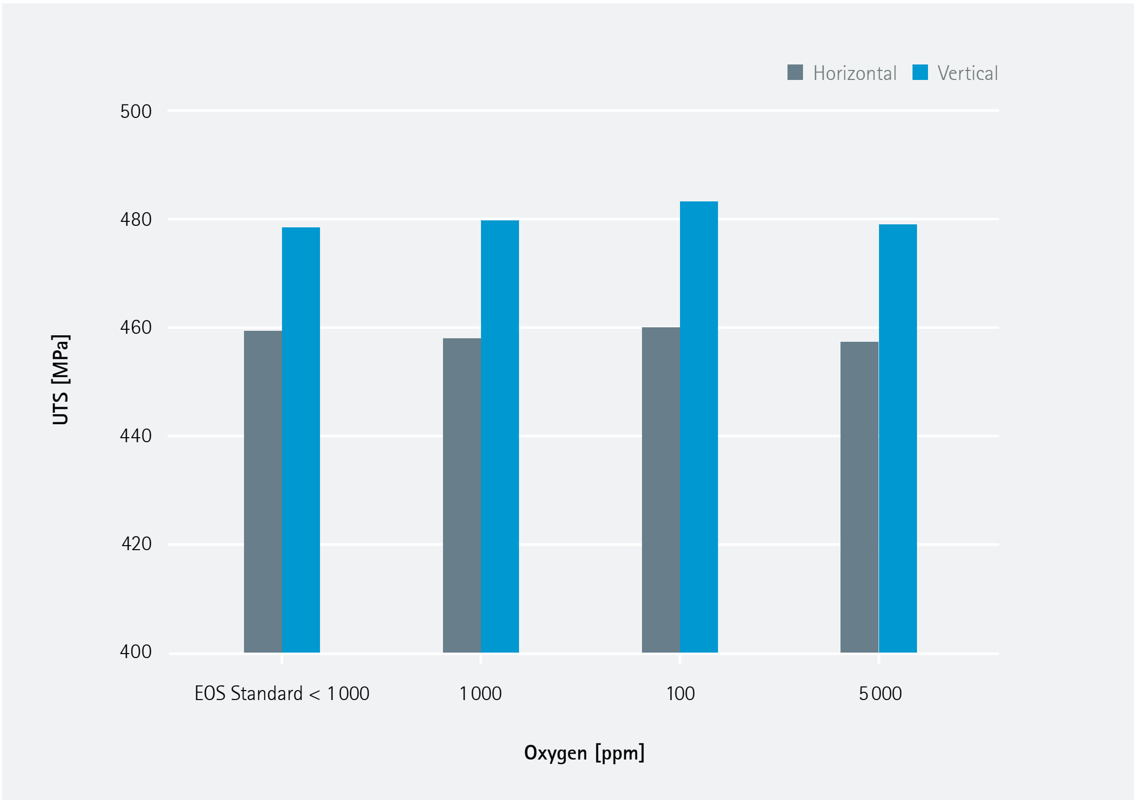

Figure 6: Ultimate tensile strength results

[2] EOS material datasheet: UTS hor.: 460+-20 MPa vert.: 470 MPa+-20MPa; YS hor.: 270+-20MPa vert.: 230+-20MPa; Elongation hor.: 10+-2% vert: 6+-2%

Fatigue Testing

Figure 7 shows fatigue results for three process conditions performed with: 1) EOS standard process gas atmosphere, 2) 1 000 ppm and 3) 100 ppm residual oxygen (2 and 3 controlled by ADDvance O_2 precision). The scatter bands (blue, green and red) are designed with the assumption that the limit of endurance is reached for the lowest maximum stresses. The fatigue results show that an oxygen level of less than 1 000 ppm ensures a high fatigue strength.

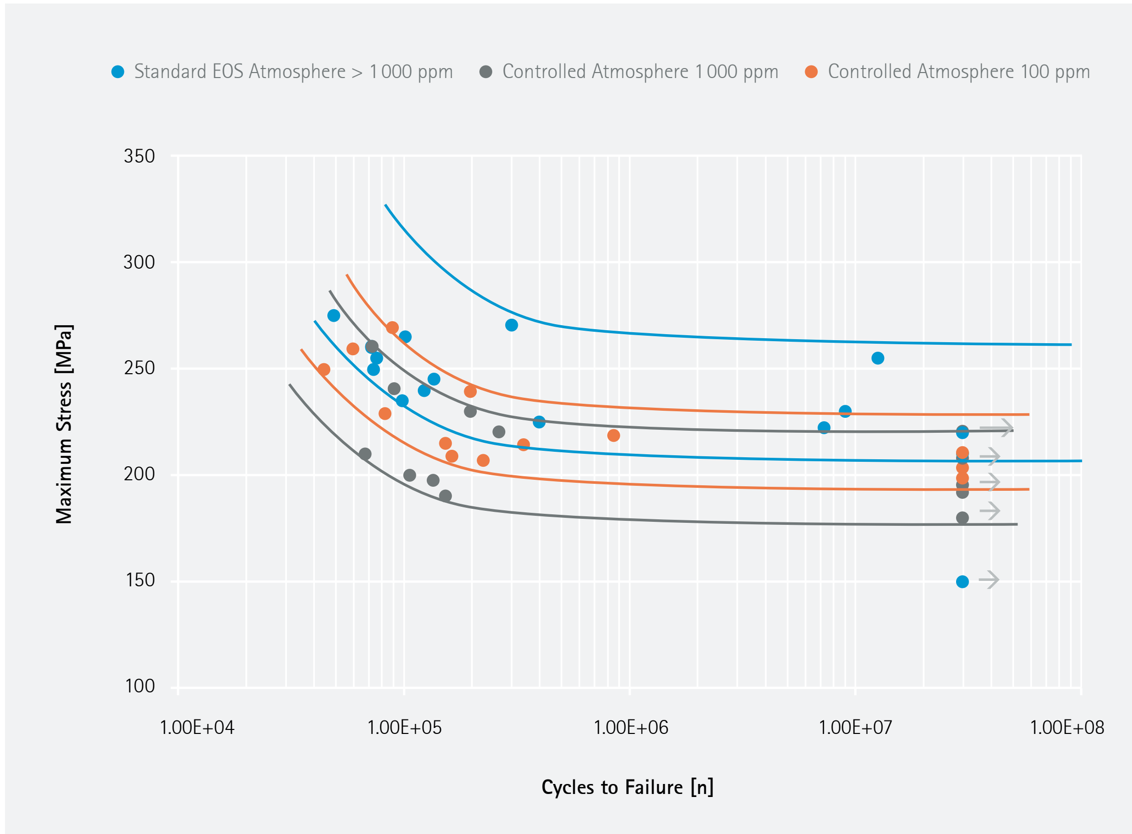

Figure 7: Fatigue results for specimens

Employing Gases beyond Argon

There are many other gas mixtures that have the potential to further improve the process stability. As part of this initial study, the influence of a special process gas mixture from Linde - consisting of argon and helium - was assessed. The same layout as shown in Figure 2 was built with standard conditions (< 1 000 ppm of oxygen) and 100 ppm of residual oxygen during the build job.

The results showed the potential to successfully produce parts with argon-helium, with some initial observations demonstrating fewer process by-products in the process chamber.

Both samples produced under argon and argon-helium show similarly high mechanical properties. The ultimate tensile strength of all four conditions has values between 470 and 473 MPa, with low deviation, as seen in Figure 8. The yield strength of the specimens is 2 % higher, while the elongation at break stays at the same level.[3]

The mean porosity value for all four conditions remains between 0.034 and 0.036 %. The maximum defect size is reduced as the oxygen level decreases.

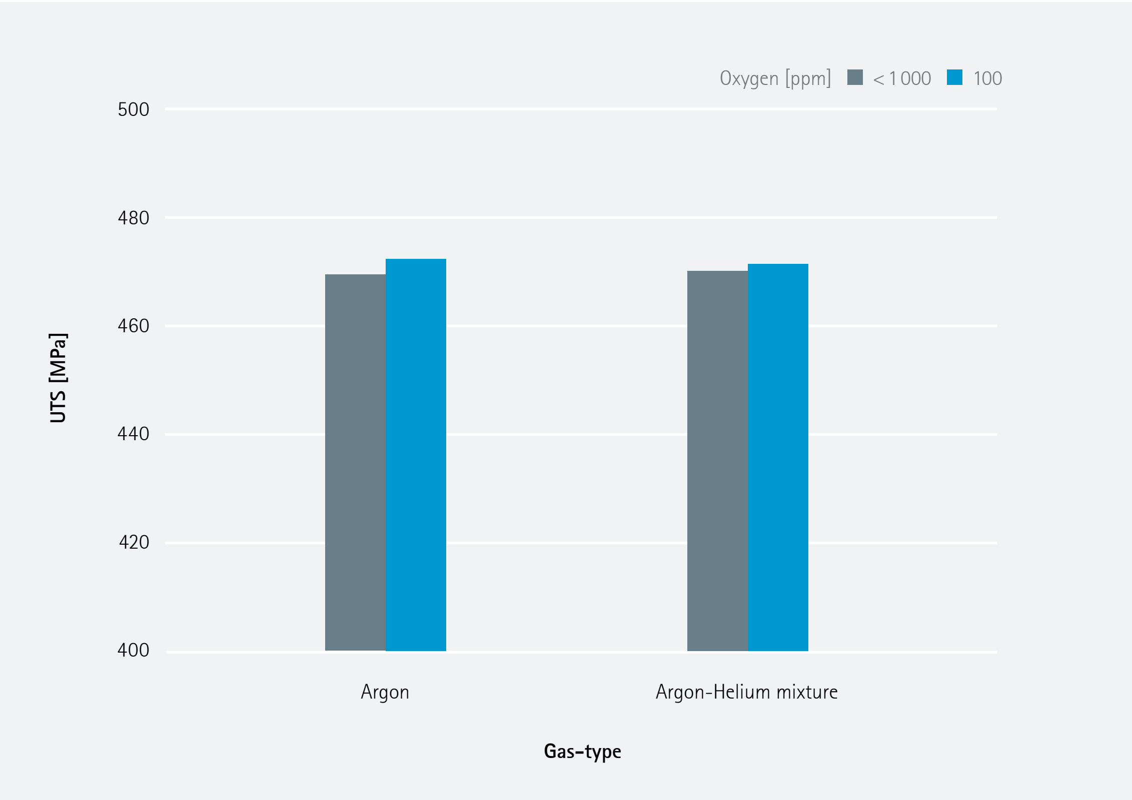

Figure 8: Ultimate tensile strength of AlSi10Mg for two different oxygen levels and two different gases

Optical Tomography Monitoring

EOSTATE Exposure OT was used to monitor the process behavior and the amount of process by-products emitted. Figures 9a and 9b show four cubes processed with argon (a) and argon-helium (b) with the same laser parameters. The cubes processed with argon-helium emit significantly fewer process by-products then those processed with argon alone. Also, the size of these by-products is smaller with argon-helium than with just argon. Fewer process by-products can lead to fewer defects, allowing the overall printed material quality to be improved. This is characterized by a clean process chamber after the build job, leading to a better powder life cycle.

These results coincide with observations previously made by Linde with Schlieren imaging.

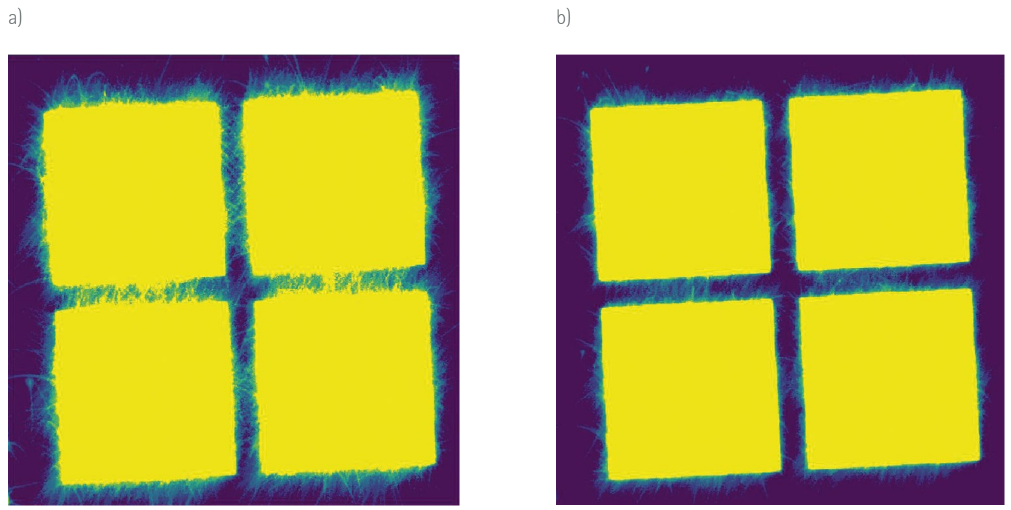

Figure 9: EOSTATE Exposure OT picture. 4 cubes with a) argon b) argon/helium process gas mixture

3 EOS material datasheet: UTS hor.: 460+-20 MPa vert.: 470 MPa+-20MPa; YS hor.: 270+-20MPa vert.: 230+-20MPa; Elongation hor.: 10+-2% vert: 6+-2%

Conclusions

The influence of the residual oxygen in the process chamber and its effect on density, tensile and fatigue properties were studied, as well as the optimal position of the oxygen sensor. The results highlight that:

- An oxygen content of less than 1 000 ppm needs to be retained during processing to ensure a very high part density. The amount and size of pores will change if the oxygen content varies between 10 and 1 000 ppm, but this does not have a significant impact on the mechanical properties.
- The EOS M 290, EOS Aluminium AlSi10Mg and EOS' AlSi10Mg process parameters enable a high-quality final part.
- An oxygen content of more than 1 000 ppm will lead to a loss of density and mechanical properties and needs to be prevented.
- Powder aging is reduced by keeping the oxygen level below 1 000 ppm, which enables more frequent reuse of the powder.
- The position of the oxygen sensor influences the measurement. A sensor placed near to the powder bed will give an optimal measurement.

Outlook

EOS and Linde will continue to combine their expertise to jointly undertake research in the field of additive manufacturing and material-gas interaction. The next study will focus on developing a new process gas for high-performance materials.

In a preliminary study, EOS monitoring and quality assurance solution EOSTATE Exposure OT was able to demonstrate that the addition of helium:

- Significantly reduces the amount of process by-products, leading to a more stable process and a lower risk of defects.
- Minimizes the fumes created in the process chamber, leading to less interaction with the laser and a more stable process.
- Keeps the same density and mechanical properties of the final part as under pure argon.

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