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Development of a machine concept for the processing of Ti-6Al-4V in the PBF-LB/M process under silandized argon atmosphere

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Abstract

The presence of oxygen in the PBF-LB/M process leads to embrittlement in the workpiece in materials with high affinity to oxygen. Especially the metal powder Ti-6Al-4V requires a special protective atmosphere during processing. By doping the argon 1.5 % with monosilane, the residual oxygen of a usual argon atmosphere is bound and reduced to a value typical for XHV (Extreme High Vacuum).
Basically, the development of an PBF-LB/M system according to VDI 2221 is presented. The admixture of silane requires an innovative machine concept in order to ensure the compatibility of the materials used and to prevent the process gases from becoming hazardous. The phases of development are accompanied by comprehensive reaction studies and flow simulations. The resulting concept relies on a compact machining area (Ø100x100mm) and breaks new ground in the processing of special materials, not only through the process gases used, but also in powder and workpiece management.
Keywords: Laser powder bed fusion; PBF-LB/M, Design; CFD; flow simulation; Ti-6Al-4V; oxygen free

1. Introduction

In the field of additive manufacturing, laser-based powder bed fusion of metals (PBF-LB/M) is a manufacturing method that has been introduced in practice in many ways. The layer-by-layer structure of the components in the powder bed leads to an expansion of the degrees of freedom in the design process. The

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process makes it possible to manufacture components with complex geometries such as internal contours and undercuts (Sapate and Apte, 2017; Klocke, 2015). In the pre-process, the component to be produced is sliced in different layers. The slices result from the position of the component in the build chamber and the selected layer thickness. During the process, a powder material is applied to a build platform, whereby the amount is determined by the layer thickness and size of the build platform. A laser beam melts the powder material according to the slice contour. The component is built up in a cycle which comprises the application of the powder material, the melting of the respective component layer and the lowering of the build platform by the layer thickness. During the build-up, work is basically done in the liquid phase of the materials (Klocke, 2015). A wide range of powder materials makes the manufacturing process increasingly attractive in the industrial environment (Sapate and Apte, 2017). On the other hand, there are limited component properties such as density, surface roughness and mechanical properties (Sapate and Apte, 2017). The process parameters, e.g. laser power, and the powder properties, such as particle size and shape, have a primary influence on these variables (Seyda et al., 2012; Santecchia et al., 2020). When using powder materials with a high affinity for oxygen, such as titanium and magnesium alloys, the residual oxygen content of the process atmosphere is an additional influencing factor (Hagemann, 2018; Dietrich et al., 2020).

The formation of oxide layers becomes a challenge during machining due to the low free surface energy and the resulting reduced wettability. Powder materials with a high affinity for oxygen are particularly affected by this as a result of the large relative surface area. Furthermore, the residual oxygen reacts with the melt during processing and leads to further embrittlement in the work piece. As a consequence, there is a great interest in increasing component quality by reducing the residual oxygen content in the process atmosphere. In addition to attempts to reduce the residual oxygen content using conventional protective gases, the approach of mixing inert gases with active gases shows enormous potential. The use of an argon atmosphere doped with monosilane was investigated first by Holländer et al. (2019). Not only does the residual oxygen react with the highly reactive monosilane, but also the moisture in the process chamber.

In terms of the properties of the components, not only the shielding gas atmosphere but also the process gas supply has a direct influence (Shen et al., 2020). A laminar gas flow directly above the laser-workpiece interaction zone is to be aimed for. Optimized gas nozzles and a free flow field within the process chamber are essential for this.

In order to investigate the significance of the above-mentioned influences on the component quality, a system concept is created in the following that enables the processing of Ti-6Al-4V powder material in an extreme high vacuum (XHV) adequate atmosphere. Furthermore, the latest developments in almost oxygen-free production and the special features of the use of monosilane as an active process gas are presented and taken into account in the concept. Despite the primary use under laboratory conditions, economic aspects are also to be included. In addition, all requirements are to meet CE certification, i.e. conformity with European safety, health and environmental protection standards. Preliminary investigations for developing this concept were already conducted by this group of authors in a separate work (Emminghaus et al., 2021). The current work therefore focusses on the derived machine components and their specific design

2. Methods

The development of the system is carried out according to the guideline VDI 2221. The VDI (Verein Deutscher Ingenieure, Association of German Engineers) is a technical-scientific association that, among various other things, writes technical rules and regulations. The design guideline divides the development process into four phases.

1. Planning and specification
   Based on the requirements for the system and the process, a functional specification is drawn up. The specifications contain all requirements that arise from a conventional additive process and are expanded by aspects from the production and maintenance of an XHV-adequate atmosphere. The technical specification forms the conclusion and transition to the next phase.
2. Concept and principle solution
   The technical specification takes over all the requirements listed in the functional specification and translates them into an optimal technical principle. In concrete terms, this means taking a different look at the assemblies of an additive manufacturing system with regard to the additional requirements due to the XHV adequate atmosphere and working out solutions for the extended list of requirements. Creative-analytical techniques such as the morphological box and utility analysis are used to evaluate and determine the optimal technical principle. Furthermore, a control layout and a safety concept for laser operation and the handling of toxic gases result from the system conditions described in the functional specification.
3. Design
   The overall design of the machine is developed taking into account technological and economic aspects. The previously determined concepts are checked and coordinated with each other in the preliminary design review. Accompanying this, a failure mode and effects analysis (FMEA) is carried out, whereby possible system faults can be identified and evaluated in the concept phase and preventive measures can be determined to avoid them. The risk assessment through an FMEA is another important element in the technical documentation in order to obtain CE certification after project completion. The final step is a complete technical design including a fully comprehensive risk assessment of the machine.
4. Realization
   The final phase of the development process involves the design of the assemblies and individual components. For this purpose, a digital mock-up is created in compliance with all previously defined functions and requirements. The design is finally released for production via a critical design review.

In order to determine possible risks and to better assess functions and requirements, investigations were carried out in advance by Emminghaus et al. (2021). The results were included in the FMEA and have a direct influence on the machine concept.

2.1. Inertisation and gas flow

Since the inerting capacity of powder materials by argon doped with monosilane has not yet been researched, preliminary tests with Ti-6Al-4V powder material were carried out by Emminghaus et al. (2021). Thereby, two different sample volumes (22.5 ml and 500 ml) were exposed to the monosilane atmosphere. The analysis of the samples under the scanning electron microscope (SEM) and an EDX (energy-dispersive X-ray spectroscopy) measurement provided information about the degree of contamination by silicon dioxide (SiO_2).

In the functional specification, limits are defined that result in a stable process and thus good component quality. For this, it is important that the gas flow runs laminar directly above the laser-workpiece interaction zone. Furthermore, gas circulation within the process chamber should prevent areas with increased residual oxygen content (e.g. undercuts). In order to match the design of the system in such a way that the requirements from the functional specification lead to the desired gas flow, a simulation was carried out by the same work group (Emminghaus et al., 2021). Since the gas supply and parts of the process chamber arise directly from the results of this simulation, the boundary conditions are given here very briefly in Table 1.

Table 1: Boundary conditions for simulation (Emminghaus et al., 2021)

Volumetric flow rate	200 l/min
Pressure	1013.25 hPa
Temperature	300 K
Gas / gas model	Argon, incompressible
Turbulence model	LES-WALE (Large Eddy Simulation, Wall-Adapting Local Eddy-viscosity)
Wall functions	kqRWallFunction, nutkWallFunction

Figure 1 shows the simplified CAD model on which the simulations were carried out.

3. Results

Basically, all the requirements that currently describe the state of the art and are implemented by industrial PBF-LB/M systems emerge from the technical specification. Some of them are essential for process reasons, such as lifting the coater during the return stroke so that the powder bed is not run over twice. Other requirements are aimed at the economic operation of the system. For example, there has to be a way to collect and recycle the unused powder material. Furthermore, the process gas should be cleaned in the machine and

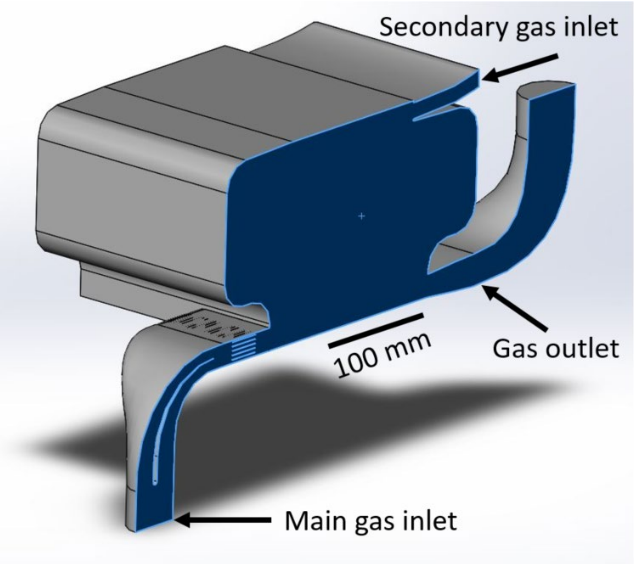

Figure 1: Simplified model of the process chamber, sectional view (Emminghaus et al., 2021)

circulate during the process. The special features result from the operation with monosilane and its strong reaction with oxygen. In the case of the powder material, it is not possible to remove it after the end of the process in a normal atmosphere, as it would be immediately contaminated by silicon-dioxide particles only several tens of micrometres in size and thus not recyclable (Emminghaus et al., 2021). The same applies to the workpiece, which results in the need for an extraction chamber in which the excess powder material and the workpiece can be removed and packed airtight under a protective gas atmosphere (argon). In the machine layout in Figure 2, the extraction chamber is shown in the middle. The process chamber is located directly above it. It is the link between the coater unit, the lifting unit and the gas-powder management.

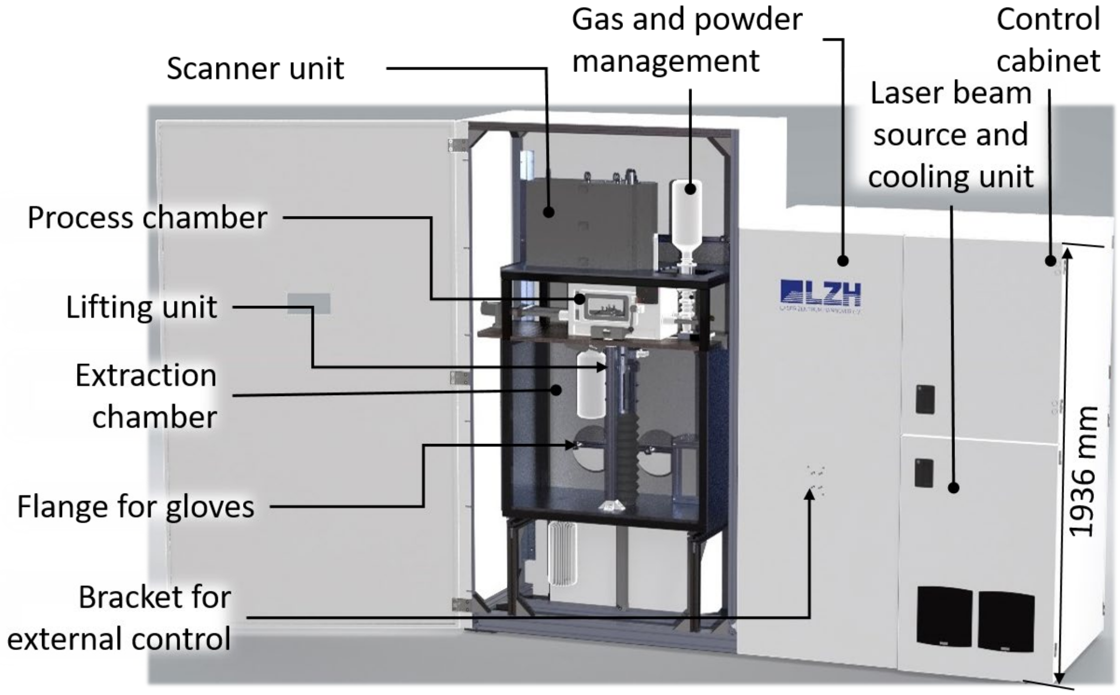

Figure 2: Schematic machine concept

The enclosure of the machine describes the laser-safe area and is CE-compliant secured. The process chamber and the gas system are considered separately in the safety control system and are continuously monitored using several sensors, such as two oxygen sensors. Depending on the system status, the sections are locked and are released, for example, by the successful back-purging of the system to argon.

3.1. Inertisation and gas flow

Based on the experiments by Emminghaus et al. (2021), the gas purging is to be realized in two stages, to achieve an atmosphere, which is similar to an XHV atmosphere with regard to the residual oxygen content. The first stage provides for inertisation with pure argon. In the second stage, the residual oxygen present is to react with monosilane to form silicon dioxide and hydrogen. The reaction equations (1) and (2) describe the relevant reactants and reaction products during inertisation:

$$SiH_4\ (g) + 2O_2\ (g) \rightarrow SiO_2\ (s) + 2H_2\ (g) \tag{1}$$

$$SiH_4\ (g) + 2H_2O\ (g) \rightarrow SiO_2\ (s) + 4H_2\ (g) \tag{2}$$

A small proportion of monosilane (10 ppm to 100 ppm) in argon is sufficient to achieve a protective gas atmosphere (Holländer et al., 2019). This led to the decision to operate the plant with 1.5 % monosilane in argon. Due to the strong reaction, it must be ensured that the monosilane can completely escape from all materials before oxygen is added. Preferably, contact with oxygen should be avoided.

The process gas is pumped into the process chamber by a side channel blower. According to the simulation, the gas is fed in through two inlet nozzles (Emminghaus et al., 2021, Figure 1). During recirculation, coarse particles are separated by a cyclone and disposed in a waste container. This is followed by fine filtration through a HEPA (High Efficiency Particulate Air) filter before the gas is fed back into the process through the side channel blower. The entire gas system and the units located in the process chamber have almost no undercuts so that they can be sufficiently flushed by the process gas and the gas can circulate freely. This is to avoid areas with gas accumulation. Furthermore, only materials that meet the requirements of monosilane compatibility are to be used. The materials were examined in preliminary tests for strong SiO_2 particle adhesion and surface changes due to the effect of monosilane (Emminghaus et al., 2021). Some plastics, such as PU (Polyurethane), showed strong surface changes (Emminghaus et al., 2021). Other materials were not affected by the active gas. In principle, it can be assumed that a rough surface increases particle adhesion and smooth surfaces are to be preferred (Emminghaus et al., 2021). Some materials, such as stainless steel 1.4401 and PTFE (polytetrafluoroethylene), are particularly suitable due to their high resistance to monosilane (Emminghaus et al., 2021).

The design of the lower inlet nozzle was optimised in three steps to achieve a laminar gas flow over the build platform (Emminghaus et al., 2021). The nozzle shape provides a slight compression of the gas from the pipe cross-section to the process chamber inlet. This compression achieves a distribution of the gas flow across the width of the build platform. The result of further optimisations shows that the implementation of a baffle in the pipe bend increases the average flow speed and a honeycomb grid at the outlet uniforms the flow (Emminghaus et al., 2021). Table 2 shows the summary results of the simulation. The different average speeds and standard deviations were measured on a parallel plane 2 mm above the building platform (Emminghaus et al., 2021). The standard deviation measures the homogeneity of the flow.

Table 2: Average speed and standard deviation (Emminghaus et al., 2021)

Simulation	**Average speed in m/s**	**Standard deviation in m/s**
Initial	1.1793	0.34856
With baffle	1.7096	0.48496
With baffle and honeycomb grid	1.1802	0.26414

The design of the process chamber is mainly derived from the simulation. The gas nozzles must be shaped according to the results and be able to accommodate the baffle plate (Figure 3, red arrow), as well as the honeycomb grid (Figure 3, red circle). The positions of the gas inlet and outlet are also determined. The purge volume of the process chamber will be less than 0.022 m^3 for economic operation of the machine. Furthermore, no components may cross the laminar flow above the building platform or create undercuts.

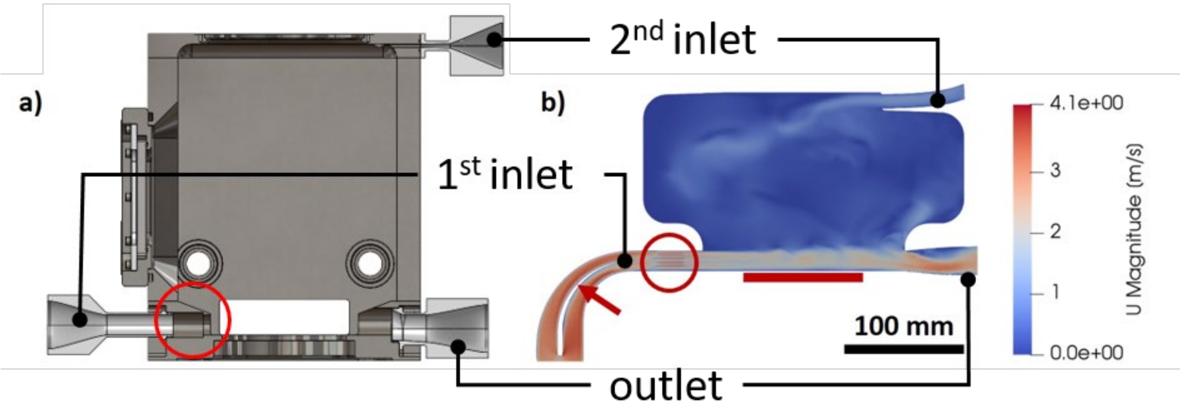

Figure 3: (a) Process chamber, (b) simplified model for simulation (Emminghaus et al., 2021)

Figure 3 shows a comparison of the simulated (b) and the derived (a) process chamber. Both models are cut in half of the build platform (position marked by red line).

3.2. Powder supply and coating kinematics

The powder material is supplied by an extruder screw (see figure 4) below the process chamber in front of the coater. In this way, complete inertisation of the powder is ensured despite the large-volume container (>0.004 m^3). The gas is fed to the unit at two points (red arrows, figure 4) and flows through the powder to the process chamber. This takes into account the results of the preliminary investigations by Emminghaus et al. (2021), according to which a small amount of powder can be inerted significantly better and contamination by SiO_2 particles can be prevented. At the same time, the large container avoids frequent refilling of powder.

The coater unit (Figure 5) is moved with up to 165 mm/s by a stepper motor located outside the process chamber. This is connected to two hollow shafts that guide the coater using linear bearings attached to the process chamber. The coater is lifted during the return stroke so as not to influence the powder bed. For this purpose, argon gas is fed through one of the two hollow shafts (red arrow, Figure 5; red arrows, Figure 6) to two mini pneumatic cylinders (see Figure 6) inside the process chamber, which lift the coater (Figure 6, black arrows indicate movement). As soon as the coater has reached its starting position, it is lowered by a spring return as shown in Figure 6. Since the shaft guides are not static, as in other systems, but part of the slide, the disadvantage is that they take up a lot of space outside the process chamber. At the same time, this arrangement has some advantages. For example, all components that are not relevant for coating, such as the motor, rotary screw, linear bearings and limit switches, can be mounted outside the process chamber and are

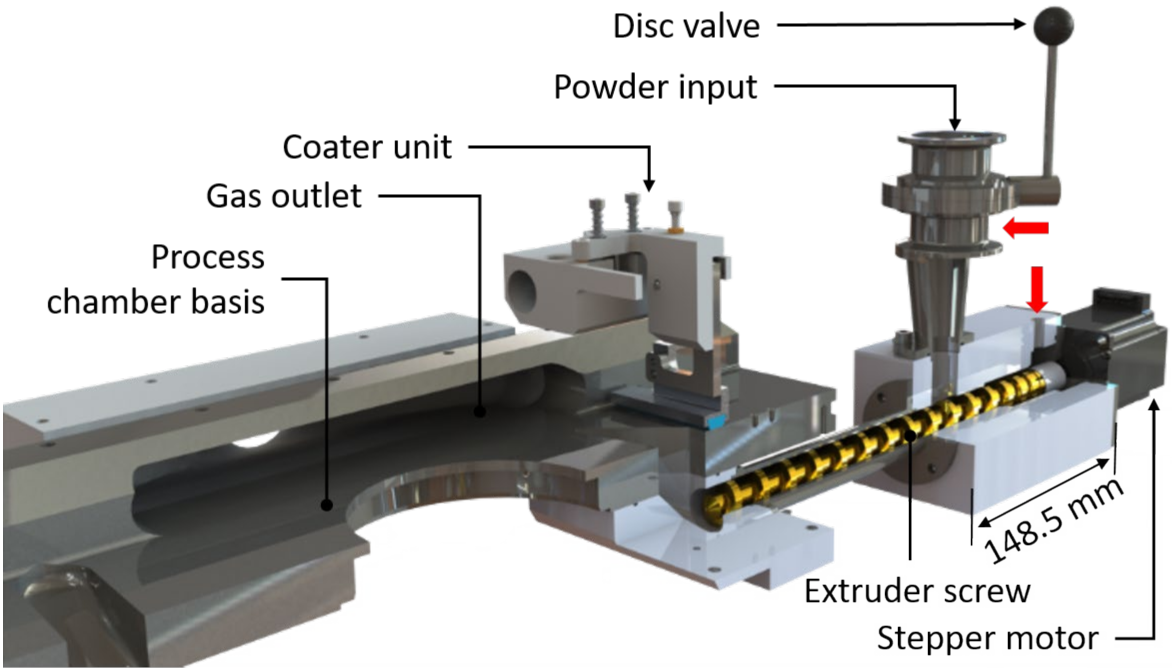

Figure 4: Powder supply unit, quarter section

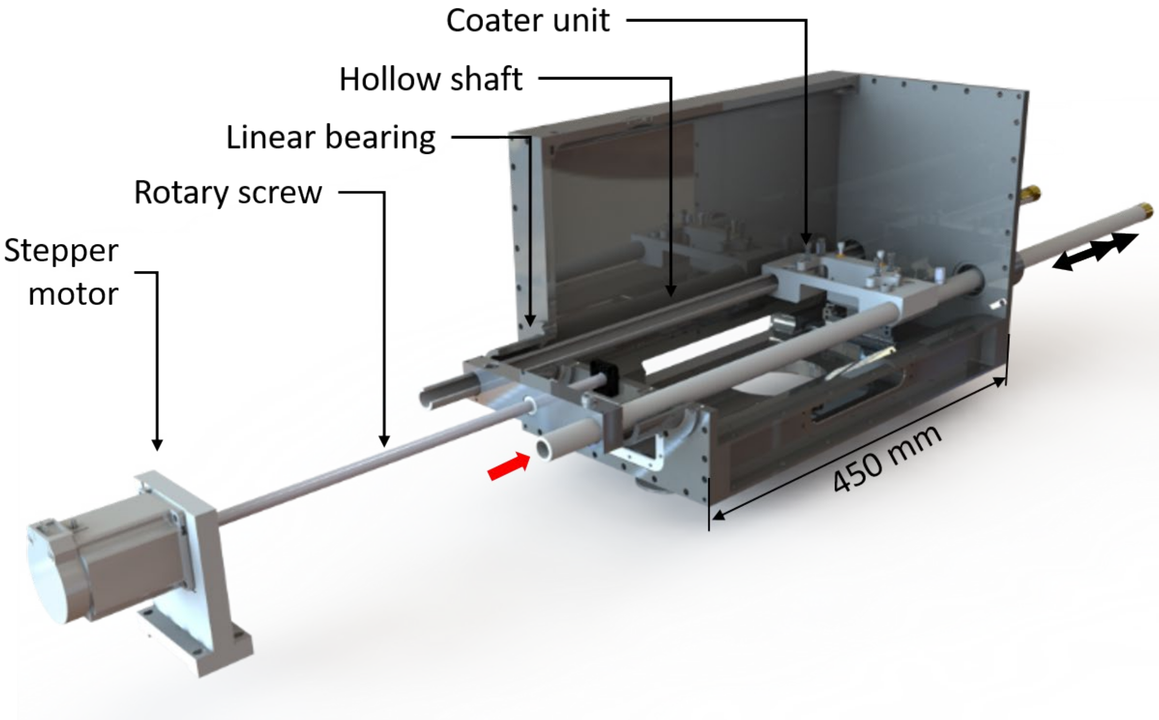

Figure 5: Coater unit; 1st hollow shaft, process chamber and two linear bearings in quarter section

not exposed to the monosilane atmosphere. Simultaneously, the number of undercuts and flow-influencing components inside the process chamber is kept low. In the case of hollow shafts, media can be fed into the process chamber without providing another opening. The process chamber is sealed at the shaft feedthroughs by using rod seals, in this case stick-slip-free PTFE seals and wiper rings.

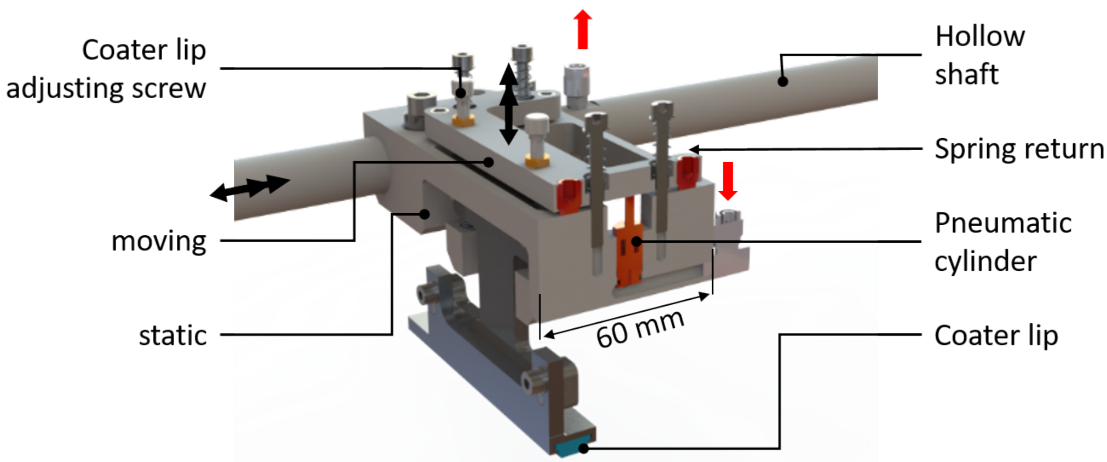

Figure 6: Coater unit, lifting mechanism. Sectional view.

3.3. Component extraction kinematics

The system concept is extended by an extraction chamber. This follows from the previously mentioned advantages, such as the airtight packaging of components. Another approach would be to expand the process chamber with openings for gloves. However, due to the required small volume of the chamber, safe handling of the components cannot be guaranteed. Since the purging time significantly influences the economic operation of the system, the protective gas atmosphere should be maintained during removal. This can significantly reduce the purging time for another subsequent process. In the extraction status, the powder bed opening to the process chamber is pneumatically closed with a lid (see Figure 7). The build platform is moved to the lowest position in the lifting unit, exposing flushing channels on the side. Argon is fed through these flushing channels on one side. A vacuum on the opposite side frees the component from loose powder material. The powder is conveyed through the channels to a cyclone, where it is separated from the gas flow and collected. Only then does the gas flow reach the pipe system with the second cyclone, the HEPA filter and the side channel blower. The entire lifting unit is then detached from the process chamber and lowered pneumatically. The building platform can thus be removed, packed or processed in the extraction chamber.

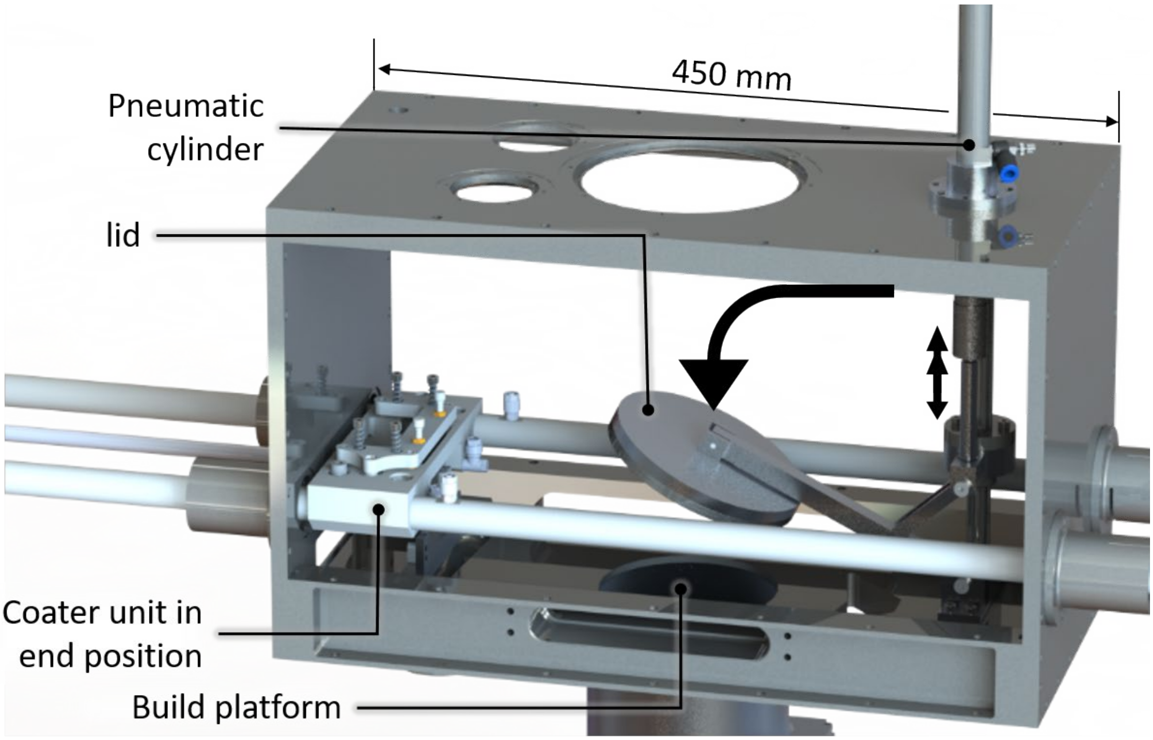

Figure 7: Closing mechanism

4. Discussion

The results of the preliminary investigations carried out have strongly influenced the machine concept and have become important for the evaluation of factors influencing the process result. By including the results in the FMEA, the assemblies could be adapted to the special requirements. Not only were the construction materials selected as a result, but the powder feed was also configured according to the inerting capacity. Since no suitable sensor for measuring the residual oxygen content was available at the time of implementation, the purging and exposure time were taken into account as influencing factors (Emminghaus et al., 2021). This consideration is poorly reproducible and leads to the fact that the required purging duration has to be determined at the machine, as this makes it possible to quantify the residual oxygen content using suitable measuring equipment and to trace it back to the process result. The results determined in the flow simulation are equally important for the design. The design of the gas system in particular has been enormously simplified as a result. However, these results need to also be compared with the actual flow in the system. On the one hand, the simplified simulation model will lead to deviations from the laminar flow. On the other hand, thermal influences caused by molten powder have not been taken into account (Emminghaus et al., 2021). The resulting assemblies are unequally complex due to the required lines of freedom and kinematics. Industrial PBF-LB/M systems pursue simpler concepts on a comparable scale. However, they offer neither the possibility of component removal while maintaining the inert gas atmosphere nor a process atmosphere adequate for XHV. After successful commissioning, extensive investigations of the process atmosphere on the workpiece properties will show whether the system concept can be justified by an increase in workpiece quality.

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