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Safety during handling of metal powders in the course of additive manufacturing: Risk assessment along the entire process chain

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Abstract

Laser powder-bed fusion is a well-known additive manufacturing variant with metallic powder. Considerable risks connected with such powder-bed processes result from the particulate nature of the raw material and from the laser-material interaction, generating respirable nanoparticles. While laser processing in closed machines is assessed as safe, preparation and post-processing of construction jobs entail an intensive contact of the operators with powders and process emission redeposits. This increases the risk of spreading hazardous substances in offices, lounges, restrooms, staircases, etc. To evaluate contamination and carry-over, the employees' exposure to hazardous substances, not only released into the air at the workplace, but also deposited on surfaces, is determined by workplace measurements for all steps of the additive manufacturing process chain. Amongst others, samples taken are analyzed using scanning electron microscopy and energy dispersive X-ray spectroscopy. Correlated with relevant assessment standards, the results shall help to derive standardized working methods for laser powder-bed fusion processes.

Keywords: laser-additive manufacturing; laser powder-bed fusion; particulate matter; residues; contamination; carry-over; occupational health and safety; process chain

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1. Introduction

Additive manufacturing processes using powdered metal materials have gained increasing importance in recent years (Campbell et al., 2018). A highly innovative process variant is additive manufacturing using laser radiation in the form of laser powder-bed fusion (LPBF). Significant risks associated with the spread of this technology occur due to the nature of the materials used for production, namely micro-scale metallic powders (primary particles) and the interaction of the laser tool with these materials. In particular, the formation of secondary particles in the form of respirable nanoparticles is to be expected (see e.g. Pohlmann et al., 2012, TRGS 528, 2009, OSHA, 2013, Gieseke et al., 2016, Schmid et al., 2016). A closed system or build chamber during proper operation, including cleaning and complete discharge of the process exhaust air to the environment, guarantees the protection of people at the workplace in accordance with the German Ordinance on Hazardous Substances (GefStoffV, 2017). However, the LPBF process with closed build chamber represents only one partial step within a multi-stage process chain. The preparation and post-processing of the actual "build job" with an open process chamber thus results in intensive contact with the powder materials and also the possibly released process emissions. There is a significant lack of knowledge concerning the risks of exposure and contamination along the process chain (see e.g. Beisser et al., 2017). In conjunction with the safety suggested with a closed build chamber, a direct consequence of this lack of knowledge is increased danger of hazardous substances being carried over into other areas, e.g. into offices and recreation rooms or even into the domestic environment of the employees involved.

For the first time, holistic process and laboratory considerations including the working environment are carried out as part of the work described here (see also Walter et al., 2019). This should help to minimize the uncontrolled release of hazardous substances and the spread of contamination. Therefore, the exposure of employees to the released hazardous substances both, in the air at the workplace (airborne hazardous substances) and on accessible surfaces (redeposits), is evaluated for various powder bed processes using metal powders. From the results of workplace measurements with regard to inhalable and alveolar dust as well as selected constituents (alloying elements), standardized working methods are to be derived by means of comparison with relevant workplace limits and assessment standards, taking into account the analysis of redeposits. Apart from work on the systems with open build area, activities during the preparation and post-processing of the build job such as filling, sieving, grinding, polishing, etc. will be considered as well.

The results of the investigations will contribute significantly to improving the protection of employees in producing companies and thus to the acceptance and dissemination of laser additive manufacturing in the form of laser powder-bed fusion in all industries along the value chain.

2. Processes, materials and analytical methods

Within the scope of the work described here, various laser powder-bed fusion processes with metal materials for the additive manufacturing of different components as well as a laser metal deposition process on a generic workpiece (reference) for comparison and general classification of the results are considered. In addition to the actual build job, each LPBF process includes various preparatory and post-processing steps, in which the processor may have contact with the powder materials used and with redeposits:

- Setting up of the system
- Additive manufacturing in the system (actual build job)
- Removal of the workpiece from the system and cleaning of the workpiece from adhering powder
- Processes for homogenizing and improving the structure (e.g. heat treatment)
- Removal of the supports for supporting the workpiece on the build platform
- Optimization of the workpiece surface and mechanical finishing (mostly manual)

Apart from the processes studied at various industry partners, an industrial LPBF process for the production of various components with an austenitic steel 316L (1.4404 according to Heinzel, 2001) was simulated at the LZH in order to study contamination with and carry-over of hazardous substances. The system used was a TruPrint 1000 of Trumpf Laser- und Systemtechnik GmbH, Ditzingen, Germany. This system comprises a fiber laser with a wavelength of 1070 nm and a maximum output power of 200 W. The system is shown in Fig. 1.

Fig. 1. LPBF system TruPrint 1000 of Trumpf Laser- und Systemtechnik GmbH used for the LPBF process simulated at the LZH.

The other powdered materials used are rolling bearing steel 100Cr6 (1.3505 according to Heinzel, 2001), cobalt-chromium alloy CoCr (according to ASTM F75, 2018), titanium alloy Ti6Al4V (3.7165 according to DIN 17851, 1990) and aluminum alloy AlSi10Mg (3.2381 according to DIN 1725-1, 1983). The raw materials were characterized by means of energy-dispersive X-ray spectroscopy (EDX), using a Fei Quanta 400F scanning electron microscope (SEM) with integrated EDX module. As an example, the element composition of the austenitic steel powder (316L), used for the LPBF process simulated at the LZH, with the main constituents iron, chromium, nickel, molybdenum and manganese according to the manufacturer's data sheet (Ecoparts, 2015) is listed in Table 1. The EDX spectrum measured for this powdered material revealed a clear correlation with the specified element composition (cf. Walter et al., 2019).

Table 1. Element composition of the austenitic steel powder (316L) used for the LPBF process simulated at the LZH, according to the manufacturer's data sheet (Ecoparts, 2015).

Chemical element	Mass fraction / %	Chemical element	Mass fraction / %
iron (Fe)	rest (main constituent)	copper (Cu)	0.50
chromium (Cr)	17 – 19	phosphor (P)	0.025
nickel (Ni)	13 – 15	sulfur (S)	0.010
molybdenum (Mo)	2.25 – 3.00	silicon (Si)	0.75
carbon (C)	0.03	nitrogen (N)	0.10
manganese (Mn)	2.0		

To characterize the emitted hazardous substances, measurements were carried out both, in the process exhaust air or circulating air in the sense of emission characterizations and in the air at the workplace in the sense of workplace analyses. An overview of the analytical methods used for this purpose is given e.g. in Walter et al., 2017. It is noted that in the context of the work described here, only the hazardous metal substances are considered in view of the basic metal materials. The main devices used for online measurements were an electric low-pressure cascade impactor (ELPI™) of Dekati Ltd, Kangasala, Finland, for determining the particle size distribution in the exhaust air or circulating air, and an aerosol monitor 8533 (DustTrak™ DRX) of TSI GmbH, Aachen, Germany (cf. Fig. 2 b and c), a device for determining four different particle fractions and total dust in room air based on the analysis of light scattering.

Fig. 2. Selection of sampling systems used (cf. Walter et al., 2019); (a) pipe with manifold for isokinetic sampling, e.g. on TruPrint systems; (b) ELPI™ (low pressure electrical cascade impactor → particle size distributions); (c) TSI DustTrak™ DRX (analysis of light scattering → 4 different particle fractions); (d) pin stub holder (capture of redeposits) → SEM/EDX, chemical analysis).

On a suitable additive manufacturing system with a sufficiently large system housing, such as a Trumpf TruPrint 3000, future investigations on the analysis of hazardous substances in the circulating air will integrate a pipe section with manifolds into the circulating air line (Fig. 2 a) to enable isokinetic sampling (cf. e.g. UBA, 2008). To date, quasi-isokinetic particle detection has been carried out on the TruPrint 1000 (Fig. 1) with a standard head for sampling of the inhalable dust fraction ("Gesamtstaub-Probenahme" – GSP 3.5, i.e. with a volume flow rate of 3.5 l/min), where the flow velocity of the protective gas in the build area is approximately equal to the suction velocity at the sampling head. The particle concentration in the build area was determined by subsequent gravimetric evaluation of the covered sampling filters.

So-called pin stub holders were used to capture redeposits (Fig. 2 d). These simple instruments can be used to scan surfaces manually. The redeposits stick to the black adhesive contact surface pressed onto the respective surface and are then covered with the plastic tube for protection. Subsequently, the samples can be analyzed chemically or with the SEM/EDX. In further investigations, cotton wool rolls were used to take wipe samples, which were evaluated by chemical analysis.

3. Inhalation exposure at the workplace and emissions in the exhaust air or circulating air

If the LPBF system is operating properly, the most intensive contact with the powder materials and the hazardous substances released from the process zone can be expected during powder filling, workpiece removal and system cleaning. When filling the system with micro-scale metal powder, however, there has been no interaction with the laser radiation. Accordingly, no respirable nanoparticles should be released if the raw material meets the manufacturer's specifications with particle diameters above 10 µm. Therefore, nanoparticles are only created during the build job by the laser radiation. Using the titanium alloy Ti6Al4V as an example, Fig. 3 shows that after opening the system during workpiece removal and system cleaning, not only a significant inhalable dust fraction but also a significant alveolar dust fraction can be detected. The absolute values for the three processes in different companies (A – C) considered here are similar. In all cases, compliance with the general dust limit value (10 mg/m³ for the inhalable dust fraction and 1.25 mg/m³ for the alveolar dust fraction according to TRGS 900, 2019) is ensured.

Stationary measurements were performed in the three companies at distances of ~ 1.2 m (local) and ~ 5 m (remote). Apparently, the dependence of the exposure on the distance of the sampling position from the LPBF system is altogether low for both, the alveolar dust fraction and the inhalable dust fraction.

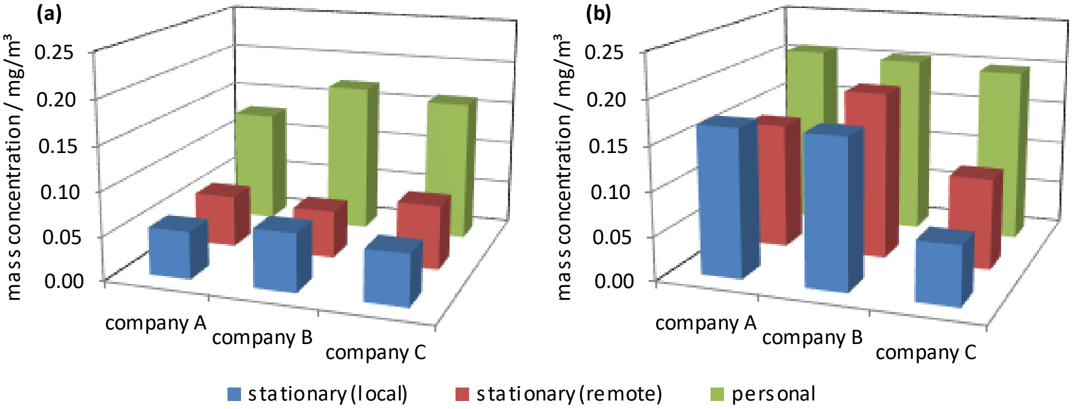

Fig. 3. Results of distance-related workplace measurements for the alveolar dust fraction (a) and the inhalable dust fraction (b) at three companies (A - C) during post-processing (removal, cleaning) at systems for additive manufacturing with Ti6Al4V (cf. Walter et al., 2019).

Compared to the stationary measurements, the particle concentrations in the personal measurements are significantly increased, especially in the case of the alveolar dust fraction. This can be attributed to the fact that the employee sometimes has to lean noticeably into the system during workpiece removal and system cleaning in order to be able to work effectively. As soon as material is whirled up during the activity, there is inevitable direct contact with airborne particles. When assessing the results, it should be noted that, on the one hand, exposure peaks may occur during the activity, which cannot be detected with the averaging gravimetric measurement method used, and on the other hand, no evaluation of the concentrations of special constituents has been carried out so far. Especially vanadium, which is contained in a significant proportion of 3.5 % – 4.5 % of the titanium alloy used (Renishaw, 2017), must be regarded as critical in this context because of the low occupational exposure limit values for vanadium(IV) and vanadium(V) compounds in the inhalable dust fraction (0.030 mg/m³) and the alveolar dust fraction (0.005 mg/m³) applicable under TRGS 900, 2019.

Direct evidence of nanoparticle formation as a result of the laser beam-material interaction during additive manufacturing can be found in Fig. 4. The images were taken from glass fiber filters in which particulate material was extracted from the exhaust air or circulating air of the process under consideration as part of isokinetic sampling. Images (a) and (b) in Fig. 4 were taken from the exhaust air of the metal deposition process used as a reference, while the filter element shown in images (c) and (d) in Fig. 4 shows material from the circulating air of the LPBF process simulated at the LZH. The magnifications depicted on the right side show quite well the large quantities of nanoparticles that were apparently formed above the process zone and have combined to form complex agglomerates of low average density.

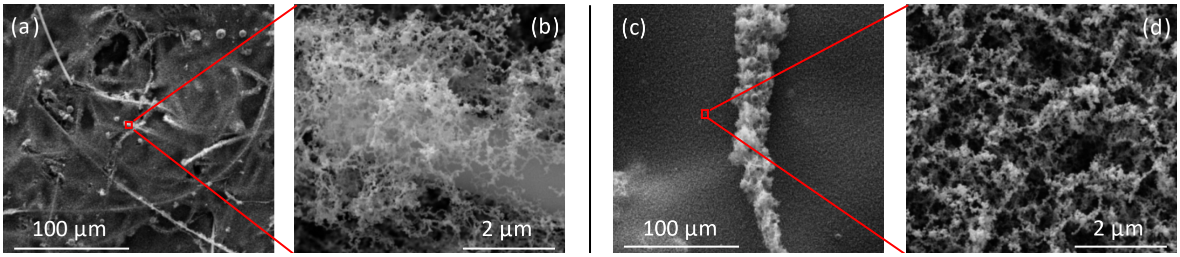

Fig. 4. (a)/(b) particle morphology from reference process (exhaust air); (c)/(d) particle morphology from LPBF process simulated at the LZH (circulating air). The fibers visible in the SEM images originate from the glass fiber sampling filters (cf. Walter et al., 2019).

Further analysis of the particulate samples taken shows that the particle sizes are within a range in which the likelihood of introduction into the pulmonary alveoli after inhalation is very high, underlining the importance of exposure-reducing measures.

4. Determination of the carry-over of hazardous substances

Due to the manual work steps required in the course of laser powder-bed fusion processes, the occurrence of contamination carry-over in the case of a non-ideal working method is obvious. This was demonstrated by the LPBF process with steel powder 316L simulated at the LZH and by the reference deposition process with steel powder 100Cr6 as well as by various LPBF processes at industry partners with different powder materials. As explained in section 2, sampling was carried out in most cases with pin stub holders with regard to hazardous substances carried over or redeposits on accessible surfaces. By repeatedly pressing on the respective contact surface at several directly adjacent points, it was possible to increase the amount of detected redeposits and consequently to lower the detection limit.

Some results of the investigations carried out at the LZH are presented below. Following a construction process with the TruPrint 1000 (cf. Fig. 1), samples were taken from various locations in the system build area. Noticeably, flaky deposits with apparently very low average density had formed on the accessible surfaces. In this case, the formation of these flakes is attributed to faulty filtering of the circulating air of the LPBF system.

SEM images of two different locations of the captured redeposits in the build area are shown in Fig. 5. The different characteristics of the redeposits are clearly recognizable. Globular particles were primarily found in the redeposits at location 1, where no black flakes were detected. In addition to particles measuring several tens of micrometers in diameter, which are predominantly present in the metal powder used, there are also many small particles measuring only a few micrometers in diameter which are apparently produced by re-condensation from the metal vapor phase or which originate from other indefinable sources. At location 2, where some of the black flakes were captured with the pin stub holder, the SEM image shows numerous globular particles with diameters in the lower nanometer range and predominantly layered, seemingly unstructured deposits. At a higher magnification (Fig. 5 c), these deposits turn out to be large, irregularly shaped nanoparticle agglomerates. This result underlines the importance of a correctly functioning filtration of the exhaust air or circulating air of LPBF processes.

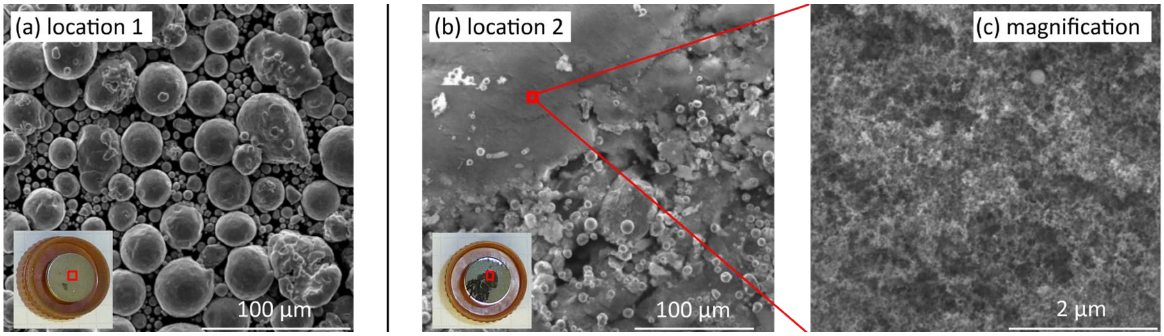

Fig. 5. Carry-over paths using the example of the laser powder-bed fusion process (additive manufacturing of components with steel powder 316L) simulated at the LZH, very different redeposits at different locations (a) and (b)/(c) in the build area of the TruPrint 1000.

A wide variety of locations for the deposit of hazardous substances are conceivable outside the processing system used or the utilized build area of the powder-bed machine. A selection is shown in Fig. 6. All pictures were taken at the LZH as examples to preserve the anonymity of the companies involved in the investigations.

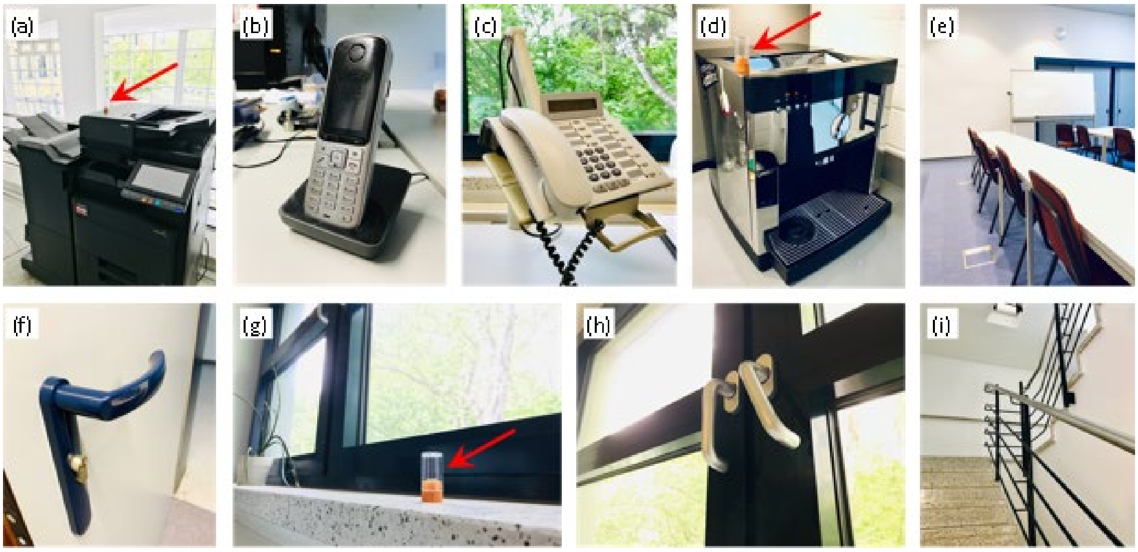

Fig. 6. Selected carry-over paths (photos taken at the LZH). (a) photocopier; (b) cordless phone; (c) landline phone; (d) coffee machine; (e) conference room; (f) door handle; (g) windowsill; (h) window handle; (i) stair railing (the arrows denote exemplary pin stub holders).

Fig. 7 illustrates the results of the SEM/EDX analysis of a sample obtained from an industry partner and taken in the way described above from the surface of a paper towel holder, located near the LPBF system under consideration (see Fig. 7 a). The towel holder was covered with a visible layer of dust at the time of sampling. The aluminum alloy AlSi10Mg listed in section 2 was processed in the LPBF system. According to the corresponding data sheet (EOS, 2014), aluminum and silicon are the only main constituents of this material, but Table 2 depicts a large number of other elements in small concentrations. Obviously, the dust captured with the pin stub holders in Fig. 7 (b) contains a significant amount of organic material, as indicated by the large carbon peak (C) in the EDX spectrum in Fig. 7 (e). Furthermore, a significant proportion of the above-mentioned aluminum alloy can clearly be identified by the peaks of the main constituents Al and Si.

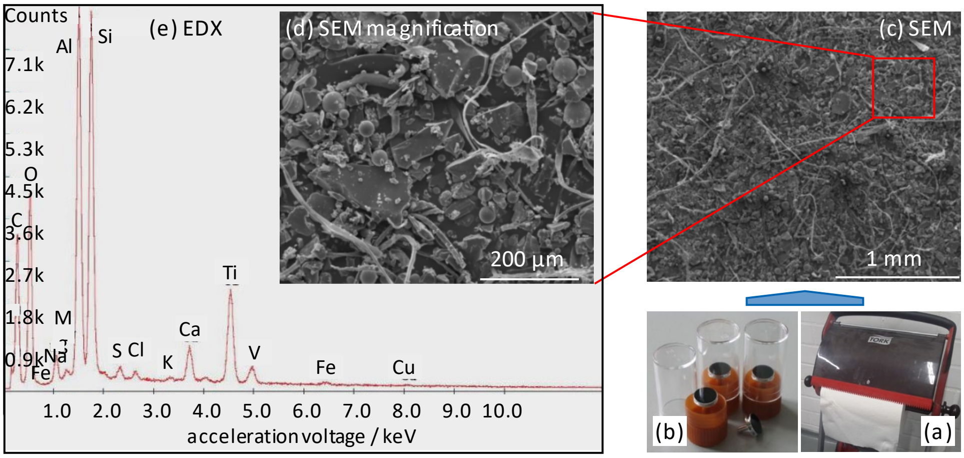

Fig. 7. Carry-over path – example towel holder (see Fig. 7 a, sampling a few meters away from the LPBF system, cf. Walter et al., 2019).

The SEM image shown in two magnifications in Fig. 7 (c) and (d) exhibits the complex mixture of particle morphologies generally expected for dust. In addition to filamentary or fibrous materials, especially irregular plate-like or flaky particles with dimensions between 10 µm and 200 µm as well as globular particles with diameters of less than 50 µm can be recognized. The latter can directly originate from the material processed in the LPBF system. Because of the minimal spatial resolution of approximately 100 µm of the utilized measuring instrument, an exact allocation of a measured element composition, determined by EDX, to a single particle is only possible to a limited extent.

Table 2. Element composition of the AlSi10Mg aluminum alloy used in the LPBF process around the towel holder sampled according to the manufacturer's data sheet (EOS, 2014).

Chemical element	Mass fraction in %	Chemical element	Mass fraction in %
aluminum (Al)	rest (main constituent)	nickel (Ni)	≤ 0.05
silicon (Si)	9.0 – 11.0	zinc (Zn)	≤ 0.10
iron (Fe)	≤ 0.55	lead (Pb)	≤ 0.05
copper (Cu)	≤ 0.05	tin (Sn)	≤ 0.05
manganese (Mn)	≤ 0.45	titanium (Ti)	≤ 0.15
magnesium (Mg)	0.2 – 0.45		

5. Assessment of risks occurring due to the deposition of hazardous substances

According to the measurement results obtained, the general dust limit value regarding inhalation exposure (cf. TRGS 900, 2019) was complied with during all LPBF processes investigated to date. Under the condition that occupational exposure limits for specific ingredients have not been exceeded, no additional protective measures with respect to inhalation exposure are necessary for the work under consideration. With the aid of chemical methods, further investigations will now be carried out to determine, to which extent garments worn during the work are likely to be exposed, in order to make a more accurate assessment of the hazard potential caused by the carry-over of hazardous substances.

Initial considerations to assess the occupational risks due to the contamination of accessible surfaces with hazardous substances have been performed. As an example, it is assumed that an employee touches with his fingertips a surface that is contaminated with metallic particles, causing the particles to stick at the finger skin. Subsequently, the employee puts a finger into his mouth, thus incorporating the particles from the fingertip. The diameter of the sticking surface of a pin stub holder amounts to 12.5 mm. The size of a fingertip is in the same order of magnitude. Thus, a further assumption is that the dust captured using a pin stub holder is a suitable measure of the amount of dust possibly incorporated with the fingertip. The SEM investigations performed so far show that the sticking surface of a pin stub holder used for capturing deposited metallic dust could be covered continuously with a layer thickness of up to 60 µm, which corresponds to larger particles originating e.g. from the raw material. For the CoCr alloy specified in section 2, the calculation yields an upper limit of the incorporated mass of 61 mg, using a material density of 8.3 g/cm³ according to VDM, 2019. Assuming an average chromium proportion in the alloy of 28 % (cf. VDM, 2019) and a typical body weight of 75 kg, the upper limit of the incorporated chromium dose is estimated to 0.23 mg/kg. According to GESTIS, 2019, the median lethal dose (LD_{50}) of referred to chromium(VI) oxide, referred to the test animal “rat” and oral ingestion, amounts to 80 mg/kg. Obviously, this LD_{50} value is much larger than the estimated upper limit of the chromium dose incorporated due to surface contamination with metallic dust via a fingertip, corresponding to a rather small toxicological risk. Moreover, it is to be expected that only a part of the incorporated chromium exists as chromium(VI). However, chromium(VI) is assessed as carcinogenic.

6. Summary

Within the scope of the work described here, initial findings were obtained in a work environment approach on contamination with hazardous substances and on the carry-over of hazardous substances to other areas in laser additive manufacturing with metals using the laser powder-bed fusion process. The actual additive manufacturing process within the laser powder-bed fusion (LPBF) process chain, carried out under typical industrial conditions, takes place in a closed system which is equipped with a suitable cleaning system for the exhaust air or circulating air. Therefore, exposure to hazardous substances is predominantly determined by the preparatory and post-processing steps such as filling the system with metal powder, removing workpieces or cleaning the system after the build job has been completed. More or less intensive contact of the employees involved with metal powders and process emissions can occur during these steps. The evaluation of the inhalation exposure in the area of the various activities carried out by employees in connection with LPBF showed that the total dust concentration is relatively low on average with a clean and careful working method. Thus, compliance with the general dust limit value according to TRGS 900, 2019, is ensured. However, on the one hand, exposure peaks occur repeatedly, for example during system cleaning. On the other hand, the increase of exposure compared to background exposure is significant, especially in the case of personal measurements. Thus, critical constituents such as nickel, chromium(VI) or vanadium(IV) and vanadium(V) can quickly exceed the corresponding occupational exposure limits. In this context, the formation of nanoparticles as a result of the interaction of the incident laser radiation with the powdered raw material has proven to be particularly relevant for the hazards that occur. If, for example, the process exhaust air or circulating air is incorrectly purified, larger nanoparticle agglomerates can enter the environment after opening the system.

The fact that the hazardous substances, released from LPBF systems, contaminate accessible surfaces and can be carried over to other areas has been proven by means of X-ray spectrometric analysis of redeposits, captured using pin stub holders. In this way, the chemical elements contained in the raw materials could be detected in various locations such as towel holders, stair railings or windowsills. Further investigations with regard to surface contamination are planned in order to significantly increase the database obtained up to this point. If employees touch the contaminated surfaces with their hands or clothing or if the employees' hair comes into contact with the surface contaminations, they will partially wipe off the redeposits and distribute them further. An estimation of the upper limit of the amount of metal dust incorporated via licking a fingertip shows that the toxicological risks are rather small. However, carcinogenic constituents may be of relevance.

The extent to which nanoparticle agglomerates, deposited on surfaces, can be whirled up, such as due to unfavorable air flow conditions, causing them to be inhaled or attached to clothing or hair, and thus carried over beyond that, will be examined in further experiments. From the obtained findings, instructions will be derived for the handling of powder materials and workpieces, contaminated with powder and process emissions, during the LPBF process chain with its various manual operations. This will help to improve the protection of employees in their daily professional and private environment.

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