Evaluating Measuring Techniques for Occupational Exposure during Additive Manufacturing of Metals
A Pilot Study

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Summary
Additive manufacturing that creates three-dimensional objects by adding layer upon layer of material is a new technique that has proven to be an excellent tool for the manufacturing of complex structures for a variety of industrial sectors. Today, knowledge regarding particle emissions and potential exposure-related health hazards for the operators is limited. The current study has focused on particle numbers, masses, sizes, and identities present in the air during additive manufacturing of metals. Measurements were performed during manufacturing with metal powder consisting essentially of chromium, nickel, and cobalt. Instruments used were Nanotracer (10 to 300 nanometers [nm]), Lighthouse (300 nm to 10 micrometers), and traditional filter-based particle mass estimation followed by inductively coupled plasma mass spectrometry. Results showed that there is a risk of particle exposure at certain operations and that particle sizes tended to be smaller in recycled metal powder compared to new. In summary, nanosized particles were present in the additive manufacturing environment and the operators were exposed specifically while handling the metal powder. For the workers’ safety, improved powder handling systems and measurement techniques for nanosized particles will possibly have to be developed and then translated into work environment regulations. Until then, relevant protective equipment and regular metal analyses of urine is recommended.

Keywords:
3D printing
additive manufacturing
industrial ecology
lighthouse
nanotracer
particle exposure

Introduction
Additive manufacturing (AM) is a term to describe a set of technologies that create three-dimensional (3D) objects by adding layer upon layer of materials. 3D technology has proven to be an excellent tool for manufacturing of complex structures in a variety of industrial sectors, such as aerospace, health care, electronics, as well as simpler end-user products (Lipson and Kurman 2013). It also has potential advantages such as that spare parts can be manufactured directly to order and that a worn structure can be rebuilt on an existing part instead of replacing the whole construction. Unfortunately, the introduction of new techniques often entails uncertainties regarding safety of the operators.

The aim of this pilot study was to use different measuring techniques optimized for different particle sizes while analyzing numbers, sizes, masses, and identities of metal particle
emissions during AM operations and work related to this process to achieve information regarding appropriate measuring techniques for optimal preventive actions.

3D printing technology has been proposed to be economically friendly (Berman 2012). In addition, AM has been projected to have positive impact on the environment by reducing energy consumption and carbon footprints, but a systemic approach to the life cycle of fabricated products is still needed (Frazier 2014).

Today, several AM techniques are available; selective laser sintering and selective laser melting (SLM) are two common AM techniques, with the main difference being if the metals are sintered or fully melted (Kruth et al. 2005). In the current study, a thin layer of metal powder mainly consisting of nickel, chromium, and cobalt (20 micrometers [µm]) was evenly added to the building plate, and then a laser fully melted the metal powder according to desired shape before a new 20 µm of metal powder was added. This process was repeated in as many cycles as needed to build the entire structure. The building of the structure was performed in an enclosed chamber filled with argon during the process. When the manufacturing process was completed, the chamber was ventilated and the surplus of powder was removed from the structure using an Ex-classed industrial vacuum cleaner. The unused powder was then filtered to remove larger particle complexes that might involuntarily have been created whereas the rest was reused in the production. The company in question had invested in eight AM machines intended for continuous manufacturing of different structures, one of them equipped with a 200-watt (W) laser and the others with a 400-W laser. The typical building time for these operations varies between 14 and 32 hours. All powder handling actions, such as filling powder to the machines’ internal storage tank, vacuuming the finished parts, and filtration of the recycled powder, are performed manually.

There are thus concerns about exposure to metal powder for the workers handling the metal powder and the AM machines. There are potential hazardous health effects of several of the metal components present in the powder, but the main concern is regarding cobalt exposure. Cobalt has been suggested to be neurotoxic (Catalani et al. 2012) and may induce cancer (Wild et al. 2009) as well as lung complications (Fontenot and Amicosante 2008; Rehfisch et al. 2012). In addition, there is a risk of generating nanosized particles during AM. Health effects of metal nanoparticles are well-known hazards in other metal processing activities such as welding (Lehnert et al. 2012; Andujar et al. 2014).

The manufacturing plant did not have a history of using the AM technique, but they did have extensive experience of metal-related productions, such as milling, welding, and grinding. As part of introducing this new technique, the company performed a risk evaluation of possible occupational hazards. Therefore, a more comprehensive investigation was requested before the construction of the planned facility, involving different printers as well as an increased number of operators.

Methods

Measurement Techniques

For measurements of nanoparticles, a Nanotracer (NT) was used (Philips, Best, the Netherlands). This instrument detects and counts ultrafine airborne particles from 10 to 300 nanometers (nm). NT carries out continuous real-time measurements and measures both particle concentration (number of particles per cubic centimeter [p/cm³]) and average particle diameters.

For measurements of airborne ultrafine particles a Lighthouse (NH) Handheld 3016 IAQ (Lighthouse Worldwide Solutions, Fremont, CA, USA) was used. It is an optical particle counting instrument, detecting airborne particles from 300 nm to 10 µm, also featuring mass concentration mode that approximates density in micrograms per cubic meter (µg/m³). In addition, Lighthouse provides up to six particle-size channels of simultaneous counting (0.3, 0.5, 1.0, 2.5, 5.0, and 10.0 µm) as well as temperature/relative humidity data.

For comparison with occupational exposure limits (total dust), traditional sampling of particles on a preweighed 25-millimeter (mm), 0.8-µm pore mixed cellulose ester filter housed inside a closed-face polystyrene cassette was performed. Personal and stationary sampling was performed during 45 minutes at a flow rate of 2.0 liters per minute (L/min). Filters were analyzed gravimetrically for total particulate mass. Metal identities were analyzed with an ICP/QMS (Thermo Fisher Scientific, Waltham, MA, USA). Because hygienic limits in Sweden are given for inhalable dust regarding cobalt, additional measures for the inhalable fraction were performed where the pump was carried by the operator.

Laser diffraction (LD) analysis of new and used metal powder was performed by the powder supplier. In brief, LD analysis measures particle-size distribution by measuring the angular variation in intensity of light scattered as a laser beam passes through a dispersed particulate sample. Large particles scatter light at small angles relative to the laser beam and small particles scatter light at large angles. The angular scattering intensity data are then analyzed to calculate the size of the particles responsible for creating the scattering pattern. The particle sizes are reported as volume % or number % depending on the aim and thereby the settings of the instrument.

Machine

The machine used for AM of metals in this study was an EOSINT M270, equipped with an ytterbium-fiber laser with a nominal output of 200 W and a chamber filled with argon during the AM process. This brand was initially selected for production because of its ability to produce high-quality products, but was not equipped with a glovebox, which will be the case in the following installations. Regardless of whether the machines are equipped with a glovebox or not, powder handling that increases the risk of particle exposure is inevitable in the current practices.
**Metal Powder**

Different components need different powder composition depending on the desired material properties. In the present process, an alloy (IN939) containing high levels of cobalt, chromium, and nickel was used. The sizes of the new powder particles were according to the producer within the range of 15 to 45 μm.

**Measuring Environment/Points**

NT, LH, and traditional filter-based particle mass analyses were performed in the AM area, the welding area, and, as a comparison, in the office. Locations described below represent different operations and environments that LH measurements refer to in the Results section:

Loc 1. Opening the door to the AM machine and vacuuming the building plate; Loc 2. Handling the building plate with the finished detail; Loc 3. Straining metal powder; Loc 4. Cleaning the machine; Loc 5. Filling up the machine with metal powder; Loc 6. The machine in operation; Loc 7. The machine inactive; Loc 8. Office; Loc 9. Workshop office level 2 at daytime; Loc 10. Workshop office level 2 at night; Loc 11. Workshop area (welding, etc.); Loc 12. Powder handling (same as Loc. 1 to 5), but with LH standing near the machine.

**Results**

Below, a summary of observations using NT, LH, and traditional mass-based filter analysis followed by inductively coupled plasma mass spectrometry (ICP-MS) identification of metals accumulated on filters is presented. To get a simplified overview of what has been performed, instruments and applications are shown in table 1.

**Filter-Based Mass and Inductively Coupled Plasma Mass Spectrometry Analysis**

To investigate the work environment, particles were collected on a 25-mm cellulose filter using a pumped air flow over the filter of 2 L/min. The mass of the collected particles was measured and the metal composition was then analyzed by ICP-MS, as described in the Methods section. The results of the filter analysis (total dust) are presented in table 2.

Measurements were performed at different locations (table 2) during one preparation cycle of 45 minutes, which was followed by 14 to 20 hours of self-propelled printing depending on desired product properties. Notably, under these investigations, it was only one machine running, but in the upcoming installations, there will be several operators and metal AM machines running at the same time, which may result in a different scenario.

Results from the filter-based mass and ICP analysis of total dust, and for cobalt an additional analysis for inhalable dust (data not shown), showed increased levels of chromium, nickel, and cobalt in the air in the AM area (table 2), but because it is only peak values from 45 minutes (when the worker was active) and not activities for 8 hours, which is considered a normal working day, and there were no available reference values for short-term exposures for these metals, it is impossible to refer to the Swedish regulation “AFS 2011:18” for occupational limit values and thereby draw any conclusions.

**Measurements of Particles <300 Nanometers**

According to the alloy distributor, the particle sizes were in the range of 15 to 45 μm, which exceeds the limits of what is considered inhalable, but given that very little is known regarding the character of recycled particles, measurements of nanosized particles were performed. Given the lack of hygienic limits for nanosized particles, the background levels and the levels while cleaning the machine were measured. Two NTs measuring particles in the range of 10 to 300 nm were positioned at different locations in the room (one near the floor and one near the ceiling) near the AM machine. The air extractor in the room was positioned at the ceiling, which will not be the case in future installations because the study has revealed that the metal particles generated while operating the AM machine relocated relatively quickly to the floor.

For the new installations, the system that was considered most appropriate for large-scale AM of metals has extraction at the floor, no recirculation of air, energy recovery through heat exchangers, air extraction (5 L per second per square meter), no dehumidification, and a stable temperature of 21 ± 1.5°C.

The background levels using NT were almost not detectable (data not shown), and the levels while cleaning the machine.
Table 2  Gravimetric and ICP-MS analysis of filters placed on the operator or at two different occasions, placed near the SLM printer and the strainer while building components from IN939 metal powder

|                | Placed on operator | Near printer 1 | Near strainer 1 | Near printer 2 | Near strainer 2 |
|----------------|-------------------|----------------|----------------|----------------|----------------|
| Dust           | 210               | 250            | 130            | 120            | 170            |
| Be             | <0.10             | <0.10          | <0.10          | <0.10          | <0.10          |
| Mg             | <2.0              | <2.0           | <2.0           | <2.0           | <2.0           |
| Al             | <20               | <20            | <20            | <20            | <20            |
| Ca             | <50               | <50            | <50            | <50            | <50            |
| V              | <0.10             | <0.10          | <0.10          | <0.10          | <0.10          |
| Cr             | 44                | 50             | 24             | 21             | 32             |
| Fe             | <100              | <100           | <100           | <100           | <100           |
| Mn             | 0.17              | 0.19           | 0.22           | 0.16           | 0.18           |
| Co             | 38                | 42             | 20             | 13             | 21             |
| Ni             | 99                | 110            | 53             | 48             | 71             |
| Cu             | <2.0              | <2.0           | <2.0           | <2.0           | <2.0           |
| Zn             | <2.0              | <2.0           | <2.0           | <2.0           | <2.0           |
| As             | <0.10             | <0.10          | <0.10          | <0.10          | <0.10          |
| Mo             | <2.0              | <2.0           | <2.0           | <2.0           | <2.0           |
| Cd             | <0.10             | <0.10          | <0.10          | <0.10          | <0.10          |
| Sb             | <0.10             | <0.10          | <0.10          | <0.10          | <0.10          |
| Ba             | <2.0              | <2.0           | <2.0           | <2.0           | <2.0           |
| Tl             | <0.10             | <0.10          | <0.10          | <0.10          | <0.10          |
| Pb             | <0.30             | <0.30          | <0.30          | <0.30          | <0.30          |

Note: ICP-MS = inductively coupled plasma mass spectrometry; SLM = selective laser melting; Be = beryllium; Mg = magnesium; Al = aluminum; Ca = calcium; V = vanadium; Cr = chromium; Fe = iron; Mn = manganese; Co = cobalt; Ni = nickel; Cu = copper; Zn = zinc; As = arsenic; Mo = molybdenum; Cd = cadmium; Sb = antimony; Ba = barium; Tl = ; Pb = lead; µg/m³ = micrograms per cubic meter.

were peaking at approximately 16,000 p/cm³ (figure 1a). The average sizes of the particles were between 50 and 150 nm (figure 1b). Interestingly, the highest peaks were found at floor level, and these particles were also the smallest. As a comparison (data not shown), in the welding area the levels of nanosized particles were significantly higher, oscillating between 30,000 and 480,000 p/cm³ depending on activities, whereas outdoor levels (in a rural area at no activities) were approximately 10,000 p/cm³. Our conclusion from these measurements was that the generation of nanosized particles smaller than 300 nm is very limited in the AM environment studied (figure 1a), but because the powder contains hazardous metals such as cobalt, protective equipment is, for the time being, recommended. In addition, these studies do not cover the impact of multiple recycling processes on particle sizes, which is a scenario that has to be further investigated.

**Measurements of Particles 300 Nanometers to 10 Micrometers**

Emissions in the particle range of 300 nm to 10 µm were investigated at the locations 1 to 12 described in the Methods, using an LH instrument. Measures were performed in triplicates. The LH instrument generates data in subintervals (0.3, 0.5, 1.0, 2.5, 5.0, and 10.0 µm) in numbers or recalculated for all intervals into masses. Here, the 0.3- and 10-µm ranges are presented (figures 2a and 2b and 3a and 3b), in numbers (a) and as masses (b), given that the peak pattern of this alloy seemed to be homogenous across the ranges. The resulting intervals are presented in figure S1 in the supporting information available on the Journal’s website. Figure 2a and 2b clearly shows particle emissions in the 10-µm interval during powder handling (locations 1 to 5). Figure 3a shows a similar pattern for 300-nm particles whereas figure 3b illustrates the difficulty of translating the number of nanosized particles to a mass unit. It is clear, though, that the number of particles generated by welding increases with decreasing particle sizes.

**Laser Diffraction Analyses**

Results from LH measurements during activities at locations 1 to 12 illustrated the presence of particles significantly smaller than the size range reported by the metal powder supplier (15 to 45 µm). After discussions with the company responsible for the analyses of the metal powder, an additional assay was performed including new and used metal powder with two different settings, volume (%) and number (%). In figure 4a, the size distribution of used metal powder using LD methodology is shown. The graph corresponds to the size range reported by the metal powder supplier, but the used particles are presented (figures 2a and 2b and 3a and 3b), in numbers (a) and as masses (b), given that the peak pattern of this alloy seemed to be homogenous across the ranges. The resulting intervals are presented in figure S1 in the supporting information available on the Journal’s website. Figure 2a and 2b clearly shows particle emissions in the 10-µm interval during powder handling (locations 1 to 5). Figure 3a shows a similar pattern for 300-nm particles whereas figure 3b illustrates the difficulty of translating the number of nanosized particles to a mass unit. It is clear, though, that the number of particles generated by welding increases with decreasing particle sizes.
with the instrument setting number (%) (figure 4c). Obtained data after adjusting the LD instrument confirmed the LH analyses and thereby that new metal powder may contain smaller particles than previously known and that AM operations generates particles of nanosize. Notably, despite the adjusted settings, the sensitivity of the instrument could be questioned regarding analyses of nanosized particles given that almost no particles are detected below 1 µm, which was the case using LH as well as NT.

**Personal Protective Mask and Clothing**

When it became clear that metal particles that may cause lung, as well as systemic, complications were indeed present in a size range that was possible to inhale, instructions regarding protective clothing for the operators, designed for nanoparticle exposure, were included in the safety regulations for this location. To further investigate whether the personal protective equipment (PPE) mask (Fan breathing mask Sundström SR 500, TH3, protection factor 250, with integrated P3...
Figure 2  (a) Number of particles (10 µm) measured by Lighthouse (10 seconds (s) measuring cycle) during activities at locations described in Methods. The dominating peaks originate from locations 1 to 5*, and the smaller peaks from location 12#, as a result of powder handling. (b) Particle (10 µm) masses measured by Lighthouse during activities at locations described above. The dominating peaks originate from locations 1 to 5*, and the smaller peaks from location 12#, as a result of powder handling. Note: Loc 7, 9 and 10 are in the base line and thus not indicated in the graph. Locations: Loc 1. Opening the door to the additive manufacturing (AM) machine and vacuuming the building plate; Loc 2. Handling the building plate with the finished detail; Loc 3. Straining metal powder; Loc 4. Cleaning the machine; Loc 5. Filling up the machine with metal powder; Loc 6. The machine in operation; Loc 7. The machine inactive; Loc 8. Office; Loc 9. Workshop office level 2 at daytime; Loc 10. Workshop office level 2 at night; Loc 11. Workshop area (welding, etc.); Loc 12. Powder handling (same as Loc. 1 to 5), but with Lighthouse (LH) standing near the machine. p/m³ = particles per cubic meter; s = second; µm = micrometers.

Discussion

Additive manufacturing is an important, rapidly emerging, manufacturing technology (Frazier 2014), and as in many
Figure 3  (a) Number of particles (300 nm) measured by Lighthouse (10 seconds (s) measuring cycle) during activities at locations described in Methods. The dominating peaks originate from locations 1 to 5*, and the smaller from location 12#. as result of powder handling. Note the continuous line, showing the emissions of nanosized particles from the welding area (loc 11©). (b) Particle masses measured by Lighthouse during activities at locations described above. The continuous top line illustrates emissions from the welding area (loc 11©). Note: Loc 7, 9 and 10 are in the base line and thus not indicated in the graph. Locations: Loc 1. Opening the door to the additive manufacturing (AM) machine and vacuuming the building plate; Loc 2. Handling the building plate with the finished detail; Loc 3. Straining metal powder; Loc 4. Cleaning the machine; Loc 5. Filling up the machine with metal powder; Loc 6. The machine in operation; Loc 7. The machine inactive; Loc 8. Office; Loc 9. Workshop office level 2 at daytime; Loc 10. Workshop office level 2 at night; Loc 11. Workshop area (welding, etc.); Loc 12. Powder handling (same as Loc. 1 to 5), but with Lighthouse (LH) standing near the machine. nm = nanometers; p/m³ = particles per cubic meter; s = second.

other cases regarding the development of new technologies, the main focus has been on product quality, which may result in comprised worker safety. There is a number of safety issues linked to nanotechnology that remain to be solved (Peixe et al. 2015). Even if most recent articles are focused on engineered nanoparticles, there is a significant amount of already obtained experience from activities involving unintentionally generated particles of nanosize, such as welding (Fontenot and Amicosante 2008; Rehfisch et al. 2012; Andujar et al. 2014). Therefore, it is highly important to monitor particle emissions in all possible sizes in the AM work environment and to use available information regarding protective equipment, possibly from other similar activities, until weaknesses in production practices are corrected.

A widely used method to secure metal powder quality is LD analysis (Eshel et al. 2004). LD analysis shows the size distribution of the particles in volume percent, which is a frequently used approach when analyzing the powder quality (figure 4). Interestingly, we noticed a mismatch between LD and LH data, and after consulting the supplier, changes were made in the
Figure 4  (a) Laser diffraction spectra (volume %) of used metal powder (IN939) confirming the reported particle range (15 to 45 µm) found in the data sheet for new metal powder; (b) Laser diffraction spectra (number %) of new metal powder showing a shift toward smaller particle sizes. (c) Laser diffraction spectra (number %) of used metal powder showing a clear shift toward smaller particle sizes. µm = micrometers.

settings of the LD instrument to number percent and then the smaller particles were possible to detect (figure 4). Besides that, new powder contained smaller particles that were previously unknown and a difference in size distribution between new and recycled powder could also be confirmed. Most probably, different AM techniques result in different particle properties and exposures, depending on the brand and design of the instrument, as well as the composition of the alloys. These investigations
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Figure 5 Lighthouse particle measurements (300 nm to 10 μm) outside and inside the personal protective fan-breathing mask, equipped with integrated P3 filter and prefiltet. On the x-axis, 1 to 16 refer to outside and 20 to 26 refer to the inside of the mask. nm = nanometers; μm = micrometers; μg/m³ = micrograms per cubic meter.

demonstrate the importance of using an instrument, as well as settings, that are optimized for the powder particle size range of interest while predicting possible occupational exposures.

Today, there are no perfect methods available for studies of nanosized particles in relation to health effects. The traditional filter-based mass estimations followed by element identification using ICP-MS are useful if metal origins need to be identified, but mass information is unreliable given that nanosized particles do not contribute to a relevant mass estimation or may not even end up on the filter. However, combining traditional filter-based mass estimations including metal identifications with instruments optimized for nanosized particles such as NT and LH will give improved information regarding emissions levels and time points for exposure.

The NT data resulting from the cleaning procedure of the AM machine showed that, under the current conditions, the number of particles in the range of 10 nm to 300 nm was very low compared to other working areas, such as the welding area. This is reassuring information given that it is previously known that nanoparticles are more toxic at the cellular level than larger particles (Peixe et al. 2015). Welders have long been exposed to small particles with increased risk of clinical manifestations (Andujar et al. 2014; Schaller et al. 2007), and in line with this, a method for assessing respiratory deposition of welding fume nanoparticles has recently been proposed (Cena et al. 2014). Unfortunately, the current study does not reveal whether nanoparticles are generated near the laser or melt together into larger complexes or whether nanoparticles are generated at multiple recycling processes.

Regarding particles in the range of 300 nm to 10 μm, a different scenario was found. LH analysis showed that when handling the metal powder, high levels of particles were detected (figures 2 and 3). It was also clear that at the present instrument settings and while using the IN939 metal powder, the sizes within the range of 300 nm to 10 μm followed the same peak pattern. Indications of a homogenous size distribution may be of importance given that, in the future, it may be sufficient to measure one or two representative size ranges of small particles to ensure the working environments’ safety. The LH measurements also indicate the advantage with monitoring time-dependent fluctuations by counting the number of particles, which allows that specific activities with increased risk of exposure could be identified.

Even with the information from the particle counting instruments it is not yet possible to relate to safety regulations, it indicates at which processes operators should wear personal protective equipment intended for exposures to nanosized particles. The measurements also suggest specific operations where powder handling systems are needed or could be managed by robots. Unfortunately, there are no regulations available yet regarding exposures for number of particles at specific sizes and/or elements; therefore, biomonitoring for metals in urine should be considered as a supplement to the air monitoring.

Future Perspectives

Results from this pilot study indicate that nanoparticles <300 nm are more common in welding areas than in the AM area, whereas regarding particles >300 nm, the highest peaks were found in in the AM area. These size differences may have significant impact on health outcomes and has to be further investigated. The pilot study will be followed up in the new facilities involving several different metal alloys as well as AM techniques. Measurements of particle emissions will be performed using different instrument brands optimized for different particle sizes. Clinical analyses of the operators as well as welders and office controls will be performed regarding lung, kidney, and liver function.
Conclusions

The study indicates that nanosized particles are generated during AM of metals, specifically particles larger than 300 nm, and that the operators are exposed mainly while handling the metal powder. For the workers’ safety, improved powder handling systems have to be developed and new technologies for monitoring the number of nanosized particles that can be translated into work environment regulations are urgently warranted. Until then, combining the mass-based filter analyses followed by ICP-MS for metal identification with particle counting instruments for measuring the number of nanosized particles is recommended. Based on the current study, an instrument optimized for the particle range of 300 nm to 10 µm is the most important tool for estimation of particle numbers as well as critical time points for exposure. As a complement, metals in urine should be analyzed routinely, preferably before and after working shifts to eliminate individual variations.

Acknowledgements, Conflicts of Interest, and Ethical Permission

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