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An Introduction to Ordered Powder Lithography: Process Description, Capabilities, and Initial Case Studies

by VH Hammond, M Bleckmann, MJ Holcomb, and IJ Holcomb Jr

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An Introduction to Ordered Powder Lithography: Process Description, Capabilities, and Initial Case Studies

Vincent Hammond
Weapons and Materials Research Directorate, CCDC Army Research Laboratory

Matthias Bleckmann
Bundeswehr Research Institute for Fuel, Materials, and Lubricants

Matt J Holcomb and Jim Holcomb Jr
Grid Logic Incorporated

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14. ABSTRACT Ordered Powder Lithography (OPL) is a nonlaser-based additive manufacturing (AM) technique recently installed at the US Army Combat Capabilities Development Command Army Research Laboratory. In this process, a multiple powder-feeder printhead is used to build up a part through an iterative layer deposition approach. Unlike other powder-bed methods in which only a single powder is typically used, in the OPL process each layer is composed of at least two powders: a nonreactive zircon casting sand, which acts as the mold in which the part is formed by the deposition of the chosen metal or ceramic powder. Upon completion, the entire build assembly is placed inside a sintering oven and the part is densified using the desired pressureless sintering operation. After cooling, the part is easily removed from the surrounding sand mold. From this brief description, it is clear that there are significant differences between OPL and existing AM methods such as binder-jet printing or laser powder-bed fusion. Thus, this report is intended to provide an overview of the OPL method and highlight some of its capabilities using a series of initial case studies on selected metal powders.			
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1. Introduction and Background

Despite its seemingly recent surge in popularity, the field of additive manufacturing (AM) is a well-established area of research. Indeed, since the first area of AM—stereolithograpy of ultraviolet-light-sensitive polymers—was commercialized in 1987, the field has continued to develop and expand to a broad spectrum of materials. For example, the method of selective laser sintering (SLS) that was initially commercialized in 1989 for the production of polymeric parts was transitioned in the mid-1990s to the production of metal components. Selective laser melting (SLM), a process similar to SLS in that a laser is used to produce parts from an iteratively layered powder bed, was also developed in this time frame. A third powder-bed method, known as binder-jet printing, was also introduced at this time. In this method, a polymeric binder is deposited on each layer in the desired pattern to form a "green part". After printing is completed, the green part is subjected to a debinding and sintering treatment to produce the fully dense part. Finally, in 1998, the metal AM arena was further expanded when Optomec Inc. (Albuquerque, New Mexico) introduced a commercial laser-engineered net shaping (LENS) machine for metal powders based on developmental work by Sandia National Laboratories. This method differs from SLM in that powder is deposited coaxially to a stationary laser head as the build table is moved to form the part.[*]

Not surprisingly, the potential to produce near-net-shape components from powder materials has attracted a great deal of interest at the US Army Combat Capabilities Development Command (CCDC) Army Research Laboratory (ARL). In the Metals Branch of the Weapons and Materials Research Directorate, this interest focused on determining which AM method is best suited for use with the custom powders of interest within the branch. For example, one system is a hydrided Ti-6Al-4V (titanium alloy) in which it was demonstrated that sintering of the powder metal can yield properties equivalent to wrought parts.[2–4] Additional systems include a nanostructured copper-based alloy as well as an oxide dispersion-strengthened (ODS) iron-based alloy.[5–7] Both of these systems display mechanical and physical properties that well exceed comparable alloys as well as excellent thermal stability. Indeed, it is their excellent thermal stability that requires the use of extrusion and/or hot isostatic pressing (HIP) to produce parts rather than conventional pressureless sintering.

To date, a series of initial efforts has been attempted for different powder–method combinations. For example, binder-jet printing was tried with the hydrided Ti-6Al-4V powder. Although a simple shape (e.g., a thin square) could be produced, there was appreciable concern for contamination during the burn-out treatment due to the strong tendency of titanium to adsorb interstitial impurities. This was especially the case for complex shapes in which the outward diffusion of binder constituents could be a complicated process. The inability to sinter the copper and ODS iron (Fe) alloy to density necessitated the use of laser-based AM methods. However, at present it is still quite

[*] This brief timeline of metal AM evolution was adopted from the 2016 report "History of Additive Manufacturing" by Wohlers and Gornet.[1] The reader is referred to this report if more detailed information is desired.

challenging to use laser AM with copper due to its intrinsic reflectivity of the laser energy, which can damage the laser head. Although a small sample of the ODS Fe alloy was produced using a LENS-type machine, the sample showed a significant drop in hardness relative to an extruded sample. In addition, the sample showed a high degree of porosity, which was attributed to poor flow of the powder through the printhead. Although brief, these attempts clearly indicate the selection of an appropriate AM method for a given powder can be a challenging process.

As a suitable AM method was not determined through these efforts, the visit to the CCDC Army Research Laboratory by a small business (Grid Logic, Inc., Auburn Hills, Michigan) to highlight a new AM method was of great interest. Specifically, Grid Logic was promoting an all-powder, non-beam based method denoted as Ordered Powder Lithography (OPL). Following Grid Logic's visit, as well as a series of follow-up discussions, it was thought the OPL method offered potential advantages for the powders mentioned previously as well as commercial powders. This belief was further confirmed by a series of small-scale processing efforts conducted by Grid Logic at its corporate facility. As a result, the decision was made to acquire an OPL system for in-house process development. This report provides a brief overview of the OPL method followed by a short selection of initial efforts that demonstrate the potential of this approach.

2. OPL: General Process Description

OPL is an all-powder-based, nonbeam AM technology. While similar to the more well-known methods in that a component is built from a 3-D model in a layer-to-layer basis, it differs significantly in how these layers are constructed. For example, in methods such as laser–powder-bed fusion or binder-jet printing, the part is constructed by either the selective melting or binder deposition, respectively, of the part geometry on each layer. In contrast, OPL uses a minimum of two powders on each layer that are deposited according to their function. The first powder, commonly called the negative powder, is typically a casting sand and forms the mold for the second, or positive, powder. It is this positive powder, which can be a metal or ceramic powder, that forms the component. Unlike the bed-based methods mentioned, the positive powder is deposited only where needed to form the component (similar to the LENS approach). Furthermore, if desired, a third powder known as the auxiliary powder can also be deposited. This powder could be used to form graded or layered structures, to achieve in-process alloying or compositing, or to serve as a fugitive placeholder that would be removed in a suitable post-densification process. Figure 1 is a still image captured during a deposition that has been labeled to indicate positive and negative powders. The OPL printhead and powder assembly are also seen in the picture, as well as the stainless-steel (SS) box used as a building container. Figure 2 shows a bar sample that was printed using the ODS Fe-based alloy. This picture shows the 45° raster pattern used to produce the infill part of the positive powder. The white "border" region is the zircon casting sand.

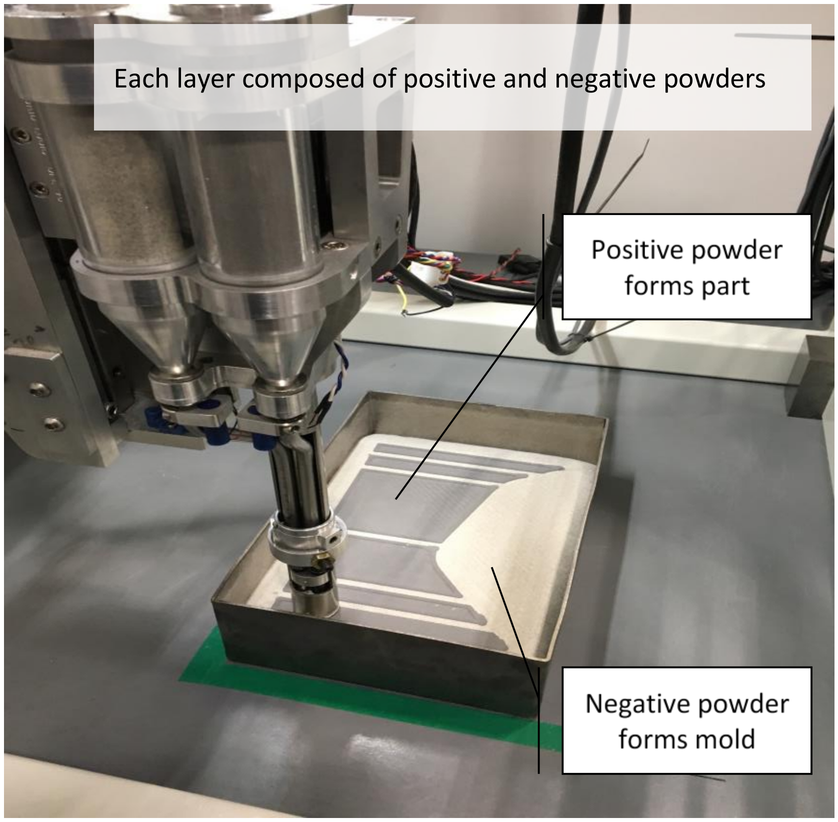

Fig. 1 **OPL deposition with negative and positive powders marked; also visible are the OPL printhead, powder hoppers, and SS box**

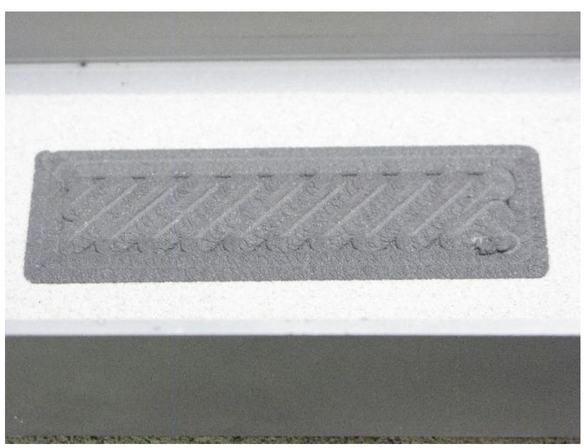

Fig. 2 **Image showing 45° pattern used for in-fill portion of a deposited layer; direction of in-fill powder is altered from ±45° on alternating layers to ensure complete filling (approximate part size = 70 × 15 × 6 mm)**

Once all layers are deposited, the entire build box is placed in a high-temperature oven in which the desired sintering process to produce high-density parts is performed. After sintering is completed, the densified part is easily removed from the negative powder (e.g., zircon sand) as this powder does not densify during the sintering treatment. In this sense, the OPL process is similar to the binder-jet process; however, unlike binder jet, OPL does not use any form of polymeric binder, so no binder burnout stage is required. Thus, OPL is able to avoid any potential contamination of the part from incomplete binder removal as well as the additional time penalty associated with binder removal. In addition, the absence of binder conceivably will allow the use of OPL to produce parts inside a container that can subsequently be converted into a HIP can for part consolidation.

3. OPL: Key Capabilities

In addition to the differences just highlighted, OPL also offers five other key "structural" features that make it an attractive AM method. First, OPL only deposits the positive powder in specific locations, thereby serving to minimize the amount of powder needed to make small test specimens suitable for sintering and subsequent evaluation. Further, this feature also serves to limit the amount of powder that is lost in a deposition due to contamination or overspill. Thus, OPL is well suited for AM efforts in which powder supply is limited, such as the in-house powder development efforts. Second, powder deposition occurs at room temperature under ambient conditions, which eliminates the need for vacuum or controlled environmental chambers. This is an important feature, as it enables several other key advantages that follow. Third, the deposition process can be interrupted after a given layer, a feature that allows for the insertion of a workpiece (such as a sensor) into the build. Fourth, the part size is limited more by the dimensions of the "gantry" that supports the printhead or by the size of the sintering furnace used to densify the part after printing than by restrictions imposed by environmental/vacuum chamber requirements. Fifth, OPL can produce samples at an extremely high rate (approximately 500 to 5000 cm^3/h), which in some cases is up to 10 times that of other AM methods.

As indicated previously, OPL can use either metal or ceramic powders as the positive powder. In the case of the OPL unit purchased by ARL, the two additional powder feeders allow for the deposition of two powders of interest as well as the negative powder on a given layer. Moreover, the OPL unit can be paused after any chosen layer, at which point a powder hopper can be emptied, cleaned, and then filled with a different powder of interest, thereby further increasing the total number of powders possible in a deposition. This capability is a significant improvement relative to powder-bed methods in which typically only one powder is used for a given deposition process. In addition, as the part created in the OPL process is densified through sintering rather than a laser melting process, there are no issues associated with process-induced microstructural changes to critical powder features (e.g., loss of nanoscale microstructures or precipitates) or compositional changes (e.g., loss of aluminum [Al] or magnesium alloying content) in the final part. Finally, as the part has been sintered to density, customary heat-treatment schedules can be used to achieve a desired microstructural state in the part (e.g., T6 for Al alloys). This is in contrast to parts produced through laser AM, in which the melting and subsequent rapid cooling results in a significantly different microstructure such that conventional heat treatments are not necessarily appropriate.

It is clear that the flowability of a given powder is a key consideration for use in the OPL process. Poor powder flow results in a poor deposit with gaps in a given layer—or no deposition at all if the printhead should clog. Indeed, in our efforts to date we have had to discontinue the use of two powders for this reason. Initial efforts suggest the powder does not necessarily have to be spherical, but must have some dimensionality to it (e.g., the powder cannot be flat or plate-like). Another key criterion for powder selection during these initial experiments was that it should be possible to achieve nearly full density through pressureless sintering. With those concerns in mind, the following are some initial case studies of using the OPL to produce relatively simple shapes.

4. Initial Case Studies

4.1 Ti-6Al-4V

As discussed previously, one of the primary powders of interest was hydrided Ti-6Al-4V alloy powder. Test specimens produced from these powders using a specialized pressureless sintering process showed mechanical properties similar to those obtained from wrought processed samples. As an initial test case, a small supply of this powder was delivered to Grid Logic for printing at its facility. The cube printed at Grid Logic is shown in Fig. 3. Two characteristics common to objects printed/sintered via the OPL process can be seen in the picture: 1) a surface reaction layer between the positive and negative powders and 2) the layered structure on the side of the part resulting from the build process itself. Both of these features can be easily removed by sandblasting or conventional milling. Possible interstitial contamination resulting from the formation of the surface reaction layer may exist, but has not yet been examined in detail.

The inward curvature observed on the sides is attributed to poor flow and powder packing during deposition. The subsequently low green density resulted in widespread shrinkage during sintering and porosity in the sintered part (micrographs confirming this are not shown). Processing efforts with this powder continued at ARL, but were eventually redirected to commercial Ti-6Al-4V powders due to the poor flow (due to their angular nature) of the hydrided powder through the OPL printhead.

Fig. 3 First cube produced using hydrided Ti-6Al-4V powder in the OPL; visible is the surface layer that typically forms during sintering, while closer inspection reveals the layered structure generated in OPL (scale of ruler is in mm)

Shifting to commercially supplied Ti-6Al-4V powder resulted in an immediate improvement in powder flow (as powder was nominally spherical) and overall part quality. Figure 4 shows the deposition of the first positive layer in a build with four bar samples. The bars are approximately 10-cm long with a 1.25-cm cross-section. Processing trials on rectangular bars yielded samples with approximately 93% theoretical density after sintering treatments. Characterization and analysis of these samples continue to determine preferred deposition and sintering protocols.

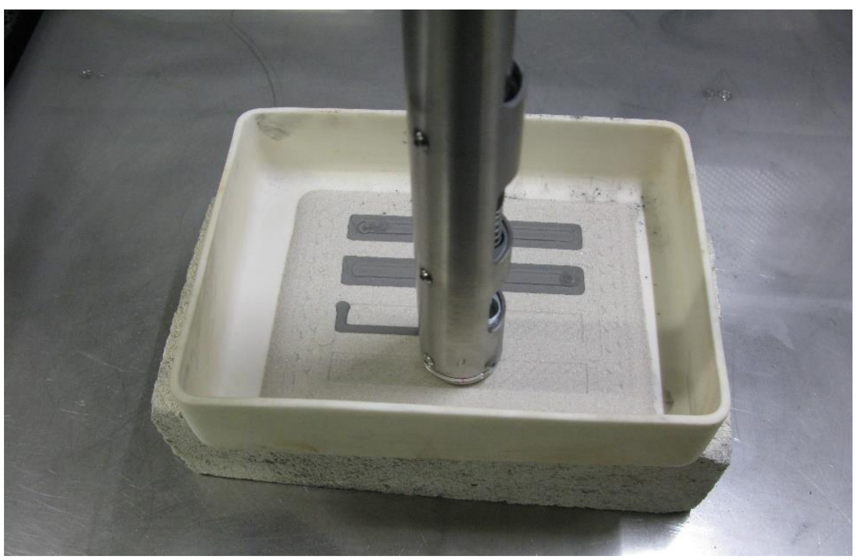

Fig. 4 Deposition of four Ti-6Al-4V rectangular bars; here, an alumina (Al_2O_3) build box is used

Once the initial standard deposition and sintering method was established for the Ti-6Al-4V powder, a more complex part was selected to demonstrate the capabilities of the OPL process. Shown in Fig. 5 is a randomly chosen turbine blade that was produced using the same protocols used for the production of the bar samples. The part is typically covered by a thin layer of sand after the deposition is completed. This layer has been brushed away and the sample partially removed from the sand so that it can be more easily seen in Fig. 5a. The build box in Fig. 5a is approximately 20-cm square. The part itself is approximately 11-cm wide by 7.5-cm tall. Density measurement indicated the part was approximately 93% of theoretical density. The individual blades are approximately 3-mm wide.

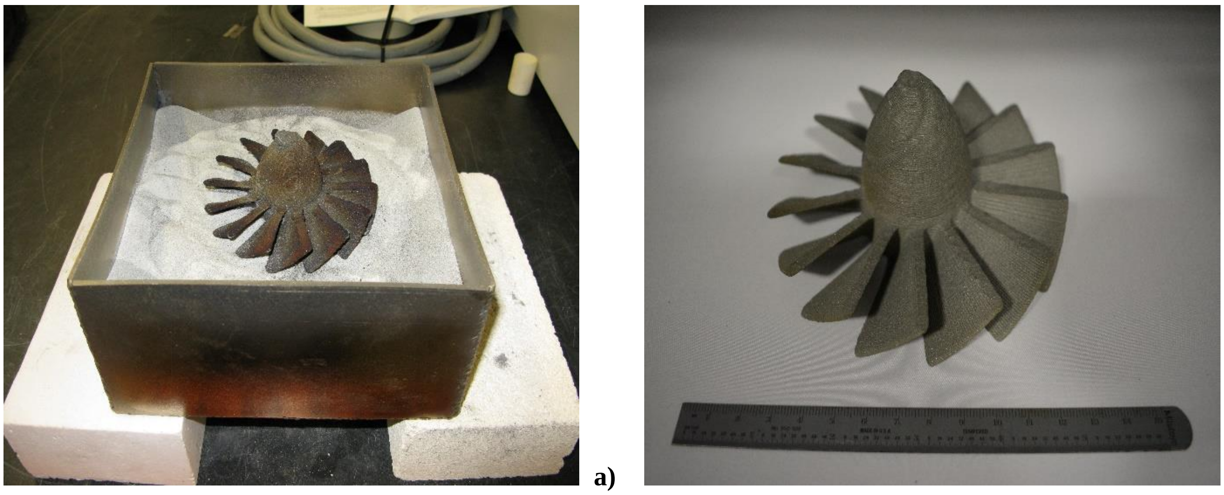
a) b)

Fig. 5 Ti-6Al-4V turbine blade produced using OPL printer and subsequently sintered at 1200 °C for 3 h; (a) blade in build box immediately after removal from sintering oven, (b) blade after removal from zircon sand

4.2 AM at Point of Need

One area where AM is expected to have an appreciable impact is in producing replacement parts at remote operating locations. In these situations, the ability to print replacement parts in a matter of hours is a significant improvement over waiting a prolonged period required for resupply. Indeed, there are appreciable efforts within ARL to evaluate the use of AM at these remote locations using powders obtained by recycling of waste materials in such locations.

In an effort to evaluate the use of OPL under these conditions, sand obtained from the Chihuahuan Desert near El Paso, Texas, was used in place of the zircon sand normally used in the OPL process. The small, rectangular test piece produced using this sand and 304 SS powder obtained by atomizing scrap plate material is shown in Fig. 6. Although the desert sand could be used in the process, it contained an appreciable amount of impurities (both organic and inorganic) that had to be removed prior to use in the deposition–sintering process.[8] This involved a lengthy acid-cleaning process followed by high-temperature pyrolysis heat treatment. The aggregated powder from this process had to be ground and sieved to the desired size limits prior to use. Moreover, the desert sand also formed a hard surface shell during the sintering process at 1200 °C that had to be chipped away to remove the sample shown in Fig. 6 (approximate size = 70 × 15 × 6 mm). Thus, although the use of a simulated indigenous sand was successful in this particular case, the use of this sand in actual operations would prove to be a significant step-down from the clean zircon sand. The use of this desert sand at a lower sintering temperature (e.g., 600 °C used to sinter aluminum-alloy powder) might be a possibility and would need to be evaluated on a case-by-case basis.

Regarding the sample, measurement indicated an approximate density of 75% for this first effort with the 304 SS powder. A second sample printed using the recycled powder and zircon sand and that used a longer dwell time at the sintering temperature was found to have a density of 93%. Efforts continue in an attempt to increase the as-sintered density of the part by using additives identified as a sintering aid for this powder.

Fig. 6 Test bar produced using recycled 304 SS powder and Chihuahuan Desert sand (scale of ruler is inches)

4.3 Iron–Nickel–Zirconium (FeNiZr) Powder

A second powder that was of initial interest in this effort was FeNiZr, an ODS iron-based alloy that was developed within the Metals Branch. An earlier attempt at producing a small sample using the LENS method was unsuccessful due to powder-flow issues. To determine suitability for the OPL method, a supply of powder was sent to Grid Logic for efforts at its facility. Figure 7 shows a "snapshot" of the deposition process in which two dogbone-type tensile bars were produced. These samples were lightly sintered to make them strong enough to handle and then sent to ARL for further sintering. However, further sintering did not result in an appreciable amount of densification. The ability to deposit a sample using this powder in the OPL was confirmed upon the arrival of an OPL unit at ARL (as shown in Fig. 2). Efforts in this area going forward will focus on the deposition of a simple part inside a HIP can, which will subsequently be consolidated using a hot isostatic press. If successful, this approach would allow the production of near-net-shape parts using powders that may otherwise not be suitable for AM approaches.

Fig. 7 Deposition of 15-cm-long dogbone tensile specimens using FeNiZr powder

5. Conclusion

OPL is an intriguing new approach to AM. While similar to many current methods in the layer-by-layer creation of a part, OPL uses an all-powder approach in which a nonsintering casting sand creates a pattern in which the powder of interest is deposited to create the part. After the deposition is completed, the entire assembly is sintered in a high-temperature oven, resulting in the densification of the deposited part. After sintering, the part is easily removed and cleaned prior to characterization and analysis.

Due to the nature of the process, OPL offers the ability to print layered parts as well as parts with inserts or selective in-situ compositing. In addition, the use of a fugitive third powder enables the production of casting molds or parts with desired internal channels. Finally, and most importantly, the absence of binders or laser melting in the OPL method offers a possible path to near-net-shape production of parts using powders not suitable for other AM methods.

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List of Symbols, Abbreviations, and Acronyms

3-D	three-dimensional
Al	aluminum
Al_2O_3	alumina; aluminum oxide
AM	additive manufacturing
ARL	Army Research Laboratory
CCDC	US Army Combat Capabilities Development Command
Fe	iron
FeNiZr	iron–nickel–zirconium
HIP	hot isostatic pressing
LENS	laser-engineered net shaping
ODS	oxide dispersion-strengthened
OPL	Ordered Powder Lithography
SLM	selective laser melting
SLS	selective laser sintering
SS	stainless steel

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