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3-Dimensional inkjet printing of macro structures from silver nanoparticles

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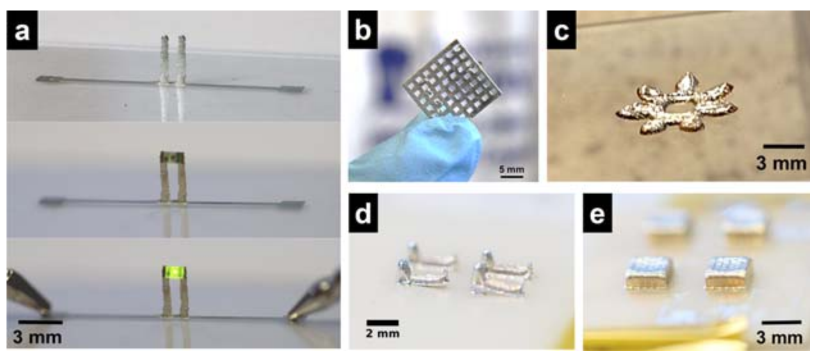
HIGHLIGHTS

- 3-dimensional structures were inkjet printed using silver nanoparticles in a single-step.
- The voids witnessed in the 600 dpi sample are primarily due to insufficient merging of the ink droplets.
- Residual surface temperature due to infrared exposure for sintering caused pinning and limited the droplets to merge.
- By increasing the printing resolution to 750 dpi, the merging of droplets was improved.
- 750 dpi sample showcased reduced void percentage and improved hardness compared to 600 dpi sample.

GRAPHICAL ABSTRACT



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ABSTRACT

The adoption of additive manufacturing technology is gaining interest for processing precious metals. In this study, the capability of inkjet printing was explored to fabricate macroscopic parts from commercial silver nanoparticle ink (AgNPs). A bespoke JETx® three dimensional (3D) inkjet printing machine was used to print and subsequently sinter up to 1000 layers of AgNPs using an infrared source. Examination of the sample using X-ray computed tomography and scanning electron microscopy revealed the existence of both micro- and nano-scale pores within the structure. Pinning effect, residual surface temperature, insufficient droplet overlap and surface defects were the key factors contributing to the voids. Elemental mapping confirmed the structure to be composed of 87% of silver along with carbon and oxygen. The 750 dpi sample showed a 25% reduction in nanopores and 77% lower micro-pores compared to the 600 dpi sample. In terms of hardness, the 750 dpi sample was 29% harder than the 600 dpi sample, showcasing samples with higher print resolution can contribute towards less voids and improved mechanical properties. Thus by demonstrating the possibility to fabricate dense parts from AgNPs using inkjet technology, this study opens a novel route for processing nano-scale particulates and precious metals in 3D.

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1. Introduction

The success of additive manufacturing (AM) in finding a place in mainstream manufacturing is primarily dictated by the added advantages of AM compared to the traditional forming and subtractive methods. Some of the key advantages of this computer aided design (CAD) driven process include, but are not limited to, (*i*) freedom of design (*ii*) the ability to produce near-net shape and end use parts, (*iii*) reduced time-to-market, (*iv*) decreased supply-chain, (*v*) reduced post-processing requirements in terms of tooling, (*vi*) high material utilisation rate and (*vii*) less wastage [1]. Among the seven AM processes classified by the American Society for Testing and Materials (ASTM), VAT photo-polymerisation, material jetting (MJ), material extrusion, powder-bed fusion (PBF) and directed-energy deposition (DED) are the most common. Among these, PBF and DED are the most widely used methods for processing metals.

With recent advancements in material development, PBF technologies, including selective laser melting (SLM), direct metal laser sintering (DMLS) and electron beam melting (EBM), are used to fabricate parts for various automotive, aerospace and biomedical applications. Although a wide range of metals and alloys can be processed using this technology, studies processing precious metals such as gold (Au), silver (Ag), platinum (Pt) and their alloys are limited [2]. There is a growing interest in the jewelry industries to use these computer driven processes to make customised products with a high degree of precision and accuracy [3,4]. Hence, AM is considered as one of the key enablers for processing precious metals.

Khan and Dickens [31] showcased the ability to process 24 carat Au using an SLM machine [5]. However, the high reflectance (85%) of the Au particles near the infrared range was observed to affect the energy absorption during laser processing, leading to poor interlayer adhesion with 12% to 15% porosity. In addition to reflectance, the thermal conductivity of Au, balling of the metals during laser scanning and spreading of the melt pool were observed to affect the SLM process [5]. SLM fabrication of dental crowns reported previously showcased a rough surface texture with inferior mechanical properties such as hardness (28 HV) and modulus (72.5 GPa) [6]. Hence in order to improve the processability of precious metals and improve the density of the fabricated parts, they are alloyed with other metals, such as silver, copper, silicon and germanium, for the jewelry applications [7,8].

Though alloying the metals may improve the SLM fabricated part's characteristics, alloying of a metal purely for processing in a particular equipment is undesirable at the expense of purity and the inherent properties of the metal. For example, the high electrical conductivity of gold may be compromised by the addition of other metals. Moreover, processing high volumes of these metals through an SLM process may add more constraints economically. With the advent of Precious M 080 by the AM machine manufacturer EOS, alloying the precious metals for SLM processing, attaining dense parts, recovering the material spread on the build platform and re-using them for future builds is no longer a problem. However, printing multiple material parts using the available PBF methods is still challenging – both technically and economically. Hence, the use of PBF for processing precious metals is less attractive.

Current research in AM has high interest in processing multiple materials to enable multi-functionality [9]. Although several AM processes, such as extrusion and MJ, have showcased fabrication of multi-material parts, it is difficult to achieve with PBF [10]. This is mainly due to the fact that separation of materials after fabrication for reuse could be problematic due to contamination of the materials.

Despite these challenges with metal AM, printing conductive tracks for electronics applications using inks containing metal flakes and metal nanoparticles (MNPs) is increasing [11]. However, in recent years, the use of inkjet (IJ) systems (a MJ technology) for printing MNPs for flexible electronics applications is extensive [12–17]. Inkjet, being a drop-on-demand (DOD) printing system, it is capable of printing at high resolution, with less material wastage. The advantage of inkjet printing has been well documented in the literature [18]. Much of the previous research concentrated on using MNPs to print circuits for electronics applications and most of them are limited to a few layers (<20) [19–21]. A recent study has documented combined printing and sintering of 50 layers of silver nanoparticles (AgNPs) towards enabling multifunctional AM (MFAM) for electronics application [22]. In concert, a recent innovation in system architecture has been shown by XJet®, whereby an inkjet based printing machine has been developed to produce metallic parts from nanoparticles [23]. The system is capable of processing a structural material and a supporting material; however, multi-material printing is limited. Microstructure, surface and mechanical properties of the parts produced by XJet® remains unknown. An understanding on the evolution of the microstructure is essential in order to build 3D structures with optimal mechanical properties. Whereas, the existing knowledge of microstructure of sintered nanoparticles for multiple layers (>100 layers) is limited.

In order to further the existing knowledge and to assess the ability of IJ printing to produce macro structures from nanoparticles, a study on printing and subsequently sintering of multiple layers of metal-nanoparticles and characterising the printed structures for porosity and mechanical stability would be beneficial. To address this, the potential of IJ printing to fabricate macro-structures from MNP inks has been evaluated in this study by printing and subsequently in-process sintering multiple layers of AgNPs using a bespoke JETx® 3D multi-material IJ printing machine. This in-process sintering mechanism can significantly save time and effort required to build multiple layers, and improve the process efficiency. The micro-structure, surface chemical analysis, porosity and hardness of the printed silver samples have been reported.

2. Materials and methods

2.1. Materials

An ink containing AgNPs (~30–35 wt%) was purchased from Advanced Nano Products, South Korea. Alumina substrates were purchased from Ceramic Substrates and Components Ltd., UK. Pre-cleaned borosilicate glass slides were purchased from Cole-Palmer®, UK. Kapton® tape was supplied by RS Components, UK and EpoFix® cold mount resin and hardener kit was obtained from Struers®, UK. Silicon carbide grinding pads and diamond pastes for polishing were supplied by Buehler®, UK. Acetone and 2- propanol for cleaning were obtained from Sigma Aldrich, UK.

2.2. Methods

2.2.1. Design

A 3 mm × 3 mm square pattern with 600 dots per inch (DPI) and 750 dpi was designed using an open source GNU Image Manipulation Program (GIMP). This design was replicated to make four 3 mm × 3 mm patterns in a 15 mm × 9 mm canvas and the image was saved as a bitmap file (.bmp). GIMP was used to design the demonstrators with a print resolution of 500 dpi.

2.2.2. Substrate preparation

Alumina substrates were used for printing the silver patterns. These alumina substrates were sonicated for 5 min using acetone and 2-propanol and placed on a glass slide. The alumina substrates were placed on the centre of a glass slide and the edges of the substrates were attached to the glass slide using Kapton® tape to prevent movement while printing.

2.2.3. 3D inkjet printing

IJP was performed using a bespoke JETx® multi-material 3D printing machine (Meyer Burger B.V., the Netherlands) using Spectra SE128