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Thin-walled high temperature alloy structures fabricated from additively manufactured polymer templates

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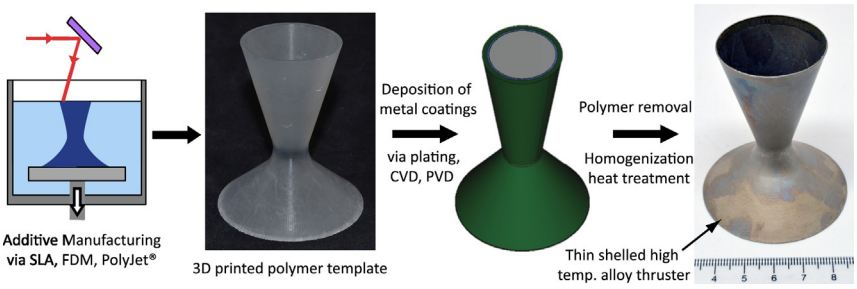
HIGHLIGHTS

- Demonstrated a new approach for indirect additive manufacturing of thin-walled high temperature alloy structures.
- Designed three alloy systems that can be formed by depositing multiple layers of metals followed by heat treatments.
- Developed a process to co-deposit rhenium and cobalt via electroplating resulting in alloys with melting points >2000 °C.
- Fabricated nickel alloy truss core sandwich structures and rocket engine thruster demos.

GRAPHICAL ABSTRACT



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ABSTRACT

Additive manufacturing is enabling a paradigm shift in design and production, but conventional techniques reach their limits when metallic structures require walls with <0.5 mm thickness. Here we introduce an approach for indirect additive manufacturing of thin-walled alloy structures. First a polymer template is additively manufactured, then metal layers are deposited by electroplating, physical or chemical vapor deposition. After removal of the polymer, the metal layers can be interdiffused to form an alloy via homogenization heat treatments. Three different alloys designed for three elevated temperature regimes are demonstrated: Monel alloys based on Ni-Cu-Al-Ti, nickel superalloys based on Ni-Cr-Al and refractory rhenium alloys based on Re-Co. A process was developed to co-deposit rhenium and cobalt via aqueous electroplating, resulting in an alloy with a melting point of 2000 °C and a Vickers hardness of 480 ± 50 $HV_{0.2}$ after homogenization. Two applications of interest for the aerospace industry were chosen to demonstrate the technology. High temperature truss core sandwich structures and rocket engine thruster demos where fabricated.

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1. Introduction

Additive manufacturing of metallic materials has created a new design paradigm which is enabling the optimization of structures far beyond any classical manufacturing method. This new paradigm is, however, not without limits. Designing thin walled structures is ultimately limited by the resolution of the available equipment. These resolutions are generally limited to around 100 μm in powder bed approaches and >500 μm in wire fed machines. While this spatial resolution of the powder bed system is extraordinary, structures produced in this size regime are ultimately weak due to the nature of

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the process [1]. Melt pool instabilities and incorporation of unmelted powder leave rough surfaces resulting in significant stress concentrators in the thin walled builds [2]. A balance needs to be struck for the powder particle size or wire diameter between layer thickness required to enable timely builds, powder flowability and stable melt pool diameter. Most commercially available 3D printers operating with powders utilize average particle sizes between 40 and 70 μm due to the industrially relevant yields of gas atomization processes and established safety protocols of working with metals powders in that size range. Increasing particle size beyond these ranges decreases particle flow, while decreasing size makes particle handling more difficult. Multiple powder particles need to be melted to initiate a melt pool, however when producing thin walled structures in conventional additive manufacturing only a small number of particles are melted with a restricted path for heat extraction, this results in the aforementioned melt pool instability and roughness [3]. A new method is needed to enable viable structures with wall thicknesses under 500 μm via additive manufacturing.

To develop this process we propose a multilevel approach to build complex alloys onto sacrificial 3D printed polymer structures. This approach builds on previous work, in which electroplating was used to create metallic microlattices [4] and truss cores [5], however through the deposition of multiple materials and subsequent interdiffusion we are able to create alloys previously un-manufacturable in an optimized architecture. This also helps overcome another hindrance of current additive manufacturing techniques, which is the limited alloy selection.

Current additive manufacturing technologies are limited to weldable alloys, and significant research is required to optimize processing conditions to produce repeatable structures and properties [6]. Even if all these criteria are met, suitable powder or wire feedstock is only available for a few alloys and difficult to produce in small batches, reducing the appeal of additive manufacturing for small production runs. By creating thin walled structures we can leverage short diffusion paths to ultimately homogenize and produce multi-component alloys with high fidelity and predictable properties. To demonstrate this new process, we have produced three different alloys designed for three different elevated temperature operating conditions.

2. Methodology

2.1. General approach

In order to produce the desired thin walled structures the process described in Fig. 1 was developed. The first step consists of producing a positive polymer template on which to deposit the thin walled structure. This can be produced through any additive polymer process including stereolithography (SLA), micro-extrusion, PolyJet® printing, fused deposition modeling (FDM), or photopolymer waveguide processing [7]. The second step is the metal deposition followed by removal of the polymer. Electrodeposition is typically chosen to apply the first metal layer(s), because of this technique's compatibility with polymer substrates and ability to deposit dense and smooth metal coatings in the thickness range of 10 μm to 100 μm in a rapid and cost-efficient manner. To enable electrodeposition, the polymer structures need to be rendered conductive, which can be accomplished by depositing an approximately 1 μm thin metal layer by electroless plating or electron beam evaporation. Once the metal coatings exhibit sufficient structural rigidity, the polymer template can be removed by chemical etching or solvent washout, depending on the type of polymer used. To this end, the polymer template has to be accessed via sufficient areas that are not coated or where the coating is removed. After the polymer is removed by etching or solvent wash out, higher temperature depositions methods can be used to apply additional layers of metals. Chemical vapor deposition processes exist for a range of metals and of special interest are low-cost pack cementation and vapor phase diffusion coating processes. Other deposition methods such as sputtering or cathodic arc deposition can also be applied. Once the coatings are built up to the desired thickness and composition, a homogenization heat treatment is applied to interdiffuse the metals and form the targeted alloy. This can then be followed by an ageing heat treatment to adjust the microstructure. Many different structures can be produced with this approach with the limits being geometric complexity and shell thickness. Since electrodeposition is not a true non-line-of-sight process, coating thickness uniformity is increasingly difficult to achieve with increasing part complexity. Slow diffusion puts a limit on the coating thickness that can be homogenized in a reasonable time, but this issue can be mitigated by depositing multiple layers.

The layered approach to multi-element deposition provides a well understood method for determining homogenizing times and temperatures. The diffusion can be modeled using the Fickian equations for diffusion along with well documented diffusivity values of the desired species. These diffusion models can be calculated prior to material production to develop a layering strategy which can optimize homogenization time and temperature. Nickel and copper with their interdiffusion coefficient of 1.41×10^{-10} m^2/s at 1000 °C [8], are highly miscible species and diffuse quickly, allowing thick layers to be applied with minimal impact on the total interdiffusion time. For example two 50 μm thick layers of Cu and Ni would interdiffuse into a Cu50Ni alloy within 24 h at 1000 °C. Co-Re alloys require a more designed approach due to the low interdiffusivity, 1.85×10^{-17} m^2/s at 1050 °C [9], of rhenium at reasonably attainable temperatures and therefore requires a different layering strategy based on multiple alternating thin layers.

While the models can be simplified to bulk layer diffusion. The layering approach also provides additional routes to improve diffusivity of the species. Depending on the deposition strategy, grain size and stress can be controlled to promote faster diffusion. For example a faster electrodeposition rate typically results in smaller grains and the stress in the deposit can be tailored with the plating bath chemistry. Decreasing the grain size increases the grain boundary area providing additional routes for grain boundary diffusion. Likewise stress in the alloy may increase the number of dislocations either typical bulk dislocations or through thickness misfit dislocations. In both cases these provide routes for pipe diffusion of species which may decrease the observed local diffusivity of species and can increase substantially from documented bulk diffusivities. This can all be modeled and selected based on the type of species being diffused and the ideal routes of diffusion in the structure.

2.2. Metal deposition

To render the 3D printed polymer parts conductive for further electrodeposition we preferred electron beam evaporation to ensure the purity of the metals deposited. Electroless nickel deposition is another technique we applied successfully, but these deposits contain 4–8 wt% phosphorous, which can be detrimental to the target alloys. Good coating coverage and thickness uniformity during electron beam evaporation has been achieved by using a rotary, 3-dome planetary fixture to continuously change the part's angle with respect to the evaporant stream, and flipping the part over halfway through the deposition. Typically a 1 μm thin Ti layer was deposited and capped with 100 nm of Pt to avoid surface oxide formation and facilitate subsequent electrodeposition. Before electroplating, the underlying polymer was exposed in certain areas by sanding off the thin metal coating, allowing access for chemical removal of the polymer at a later stage. With the rest of the surface of the part still covered by a conductive Ti/Pt layer, a power supply was connected via multiple wires to carry out electrodeposition.

Electrodeposition of nickel was conducted in a commercial nickel sulfamate bath (MacDermid Barrett) at a temperature of 49 °C, pH 4 and low current density of 50 mA/cm^2 resulting in a deposition rate of 60 μm/hour. The orientation of the part with respect to the anodes was changed regularly to maximize thickness uniformity of the coating. Custom anodes can be designed to cover hard to reach areas. Electrodeposition of copper was carried out in a commercial copper sulfate bath (Technic Elevate 40) at a temperature of 25 °C and current density of

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