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Interface engineering of functionally graded steel-steel composites by laser powder bed fusion

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Abstract

In this study, the manufacturability of new types of steel-steel composites on an open-architecture laser powder bed fusion (LPBF) system is demonstrated. A hard martensitic stainless tool steel 440C and a soft austenitic stainless steel 316L are combined in a “multi-material” component with discrete and continuous material gradients. Interface engineering is accomplished through a heat treatment to induce phase transformation. A designed distribution of carbides/carbonitrides and local martensitic transformation lead to controllable variations in hardness. Hence, the compositionally-graded component is converted into a functionally-graded composite. The concept showcases the potential for selectively engineering the properties of steel-steel additively manufactured composites.

Keywords:
Additive manufacturing
Compositionally-graded materials
Functionally-graded materials
Steel-steel composites
High-carbon martensitic steels

1. Introduction

Additive manufacturing (AM) lends increased design freedom for producing unique functional geometries [1]. As a selective consolidation process, AM offers flexibility in functionally graded materials (FGMs) [2,3]. While multi-material AM has been extensively studied in samples produced by directed energy deposition (DED) [4–6], research on multiple metallic materials using laser powder bed fusion (LPBF) is comparatively limited [7]. Compared to DED, the primary drawback of multi-material LPBF is the mixed unmelted powder, which requires precise composition analysis and powder separation prior to reuse. Nevertheless, the superior spatial resolution of LPBF makes it an attractive technology to produce FGMs. In previous work [8], an open-architecture LPBF system was used to demonstrate a multi-material 316L-MS1 steel-steel graded component with a single transitioning interface. In the present paper, a different combination of steels is studied, which includes the 316L and high-carbon 440C steels.

High-carbon martensitic steels often require high-temperature preheating to avoid crack formation during LPBF due to the stresses associated with martensitic transformation [9,10]. However, martensitic steels containing additional nitrogen, thus lowering the martensitic start (Ms) temperature, can be suitable for low-temperature LPBF as they maintain their austenitic structure (albeit metastable) at room temperature [11,12]. The combination of austenitic and martensitic steels improves the manufacturability and allows interface engineering through phase transformation achieved by heat treatment [4,13]. The current work presents, for the first time, LPBF metal–metal composite FGM parts made of the high-carbon 440C and 316L steels in as printed (AP) and heat treated (HT) conditions.

2. Material and methods

Stainless steel powders were manufactured by nitrogen gas atomization. The 316L powder was from Höganäs ($D_{50} = 42 \mu m$), and the 440C powder was from Nordic metals ($D_{50} = 37 \mu m$). The maximum elemental compositions are 316L (C 0.03, Mn 0.2, Si 1, Cr 18, Ni 14, Mo 3, N 0.1 and Fe balance) and 440C (C 1.06, Mn 1, Si 1, Cr 18, Mo 0.75, N 0.15, and Fe balance). The nitrogen and carbon were measured using a LECO TN500 and CS230 analyzer, while the other compositions were specified by the manufacturer. The AM parts were built using an Aurora Labs system with optimized parameters from the previous work by the authors [8]. The dosing accuracy of two powders in a single layer was measured to be within 1% deviation by weight. A constant laser power = 300 W, speed = 30 mm/s and layer thickness = 30 \mu m were utilized to make $10 \times 10 \times 7 \ mm^3$ coupons.

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Two types of multi-material components were investigated in the current study. The first was a discrete A-B type with one material change from 316L to 440C (Fig. 1a). The second was a continuous steel-steel composite with a sinusoidal material dosing (A5B5). The composition changed from 100% A to 100% B every five layers (Fig. 1b). Layer transitions of 1, 3 and 5 layers were investigated to evaluate the size of the bands between the compositionally graded materials. The 5-layer transition (A5B5) was selected to ensure that the material mixing during the re-melting of subsequent layers, still enabled the formation of 440C-rich (hard) and 316L-rich (soft) bands of material. The dense samples (>99%) were further investigated in the AP and HT conditions. The multi-step HT consisted of homogenization at 1130°C, austenitizing at 1030°C, gas cooling and cryogenic treatment. The crystalline phases were identified by a Bruker-D8 XRD system using Cr-Kα radiation. Three hardness profiles were measured on mechanically polished samples using a Struers-DuroScan70-G5 (100 g, 10 s). A set of samples were electropolished and investigated using EBSD in a Zeiss-Supra-35 SEM with a step size of 4 µm over an area of ~1 mm². For the microstructural investigation, the samples were etched in Kalling’s reagent for 5 s, such that SS440C was fully etched, while the SS316 was lightly etched. A Zeiss-Axio microscope was utilized to capture optical micrographs.

3. Results

3.1. Microstructure and phase variation

Dark-field optical micrographs (Fig. 1c,d) show the microstructure in the AP condition of the A-B and A5B5 samples. The discrete interface in the A-B sample is 1–2 layers (<60 µm) thick. The A5B5
sample shows a repeating structure with alternating bright and dark bands due to different etching of the two materials. The top layer during manufacturing is rather rough because of the large beam spot size (200 μm) of the CO2 laser, which results in the wavy appearance between the layers in Fig. 1d. The rough top layer affects that the material composition along the build direction and creates a variability determined by two factors, namely the programmed dosing and the surface roughness.

The heat treatment changes the AP microstructure due to the high-temperature homogenization and results in a uniform distribution of small carbides and/or carbonitrides as seen in Fig. 1e. The carbides/carbonitrides are observed primarily in the 440C-rich layers and appear to be located at the former cell boundaries, which are typically formed through elemental microsegregation during LPBF processing [14] and which decompose during further high-temperature heat treatments [15]. In the A5B5 sample, there is a gradual transition from a fine-grained precipitate-rich 440C layer (with pinned austenite grains) to a coarse-grained 316L layer. Fig. 2 shows the EBSD phase map of the A5B5 sample in the AP and HT conditions indicating a phase change in 440C rich regions during HT, with a thickness of ~2–3 layers and repeating approximately every 10 layers (300 μm).

XRD analysis shows that the 440C powder is fully austenitic (see Fig. 3), and so is the 316L powder [14]. The high carbon content together with the appreciable amount of nitrogen in the 440C powder stabilize austenite thus improving the manufacturability and produces a single-phase steel when combined with 316L. The peak broadening and asymmetry in the AP sample are likely due to microstresses and the presence of two compositionally different austenites. In A5B5-HT, a shift toward higher diffraction angles of the austenite peaks is evident, suggesting a reduction in the amount of dissolved interstitials and/or change in the stress state. Moreover, it contains pronounced martensitic peaks (~68°, ~150°) and small peaks attributed to M_{23}C_{6} carbides, resulting from the heat treatment (cf. Fig. 1e,f,Fig. 4).

### 3.2. Hardness variation

In the AP A-B sample, a gradual increase in hardness is observed across the discrete interface. The HT leads to an increase in hardness on the 440C side as a result of the martensitic transformation. In contrast, the hardness on the 316L side is reduced primarily due to the loss of the cellular substructure (see Fig. 1e,f). The gradual changes in HV across the interface in both AP and HT samples suggest an inter-mixing of the two materials during LPBF.

The A5B5-AP sample shows a continuously graded sinusoidal variation, with the higher hardness in the 440C-rich regions. The A5B5-HT sample has a significantly higher hardness than the AP condition. On average, the hardness of A5B5-HT is lower than that measured on the 440C side of the heat-treated A-B sample. Interestingly, the minimum hardness in the soft bands of the A5B5-HT sample is significantly higher than that on the 316L side of the A-B-HT sample. This is attributed to material mixing across multiple LPBF layers which could cause the 316L-rich zones to have carbides/carbonitrides.

The results demonstrate a methodology to manufacture single-phase, steel-steel composites converted through heat treatment into dual-phase FGMs exhibiting locally engineered properties. Further, a reduced spacing (down to 50–100 μm) between soft and hard bands, indicates vastly improved spatial resolution compared to DED technologies [4]. The new process chain also enables the crack-free LPBF of martensitic tool steels with higher carbon contents than previously reported multi-material austenitic-ferritic steels [6], and bi-metallic tool steels [13].
4. Conclusions

The current work presents a new route for manufacturing steel-steel soft-hard composites by LPBF. Two types of multi-material structures were manufactured with discrete and continuously varied compositional gradients between a ductile 316L steel and a hard tool steel 440C. The interfacial gradients in the AP condition (single-phase) were further enhanced through HT due to a local martensitic transformation (dual-phase). The results demonstrate the potential for interface engineering of steel-steel composites by leveraging the spatial resolution of LPBF, thus producing functional components with locally tailored geometric and material gradients.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Fig. 4. Hardness profile across the interface for the discrete (A–B) and continuous (ASB5) samples in the AP and HT conditions.