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# Influence of co-deposition strategy on the mechanical behavior of additively manufactured functionally integrated materials

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<i>Keywords:</i> Stainless steel Ni-based superalloy Metal additive manufacturing Directed energy deposition Functionally integrated materials Digital image correlation CALPHAD	The co-deposition of multiple powder feedstocks during metal additive manufacturing (AM) can be used to fabricate materials with spatially dependent properties, which can be engineered to contain different function- alities (i.e., functionally integrated materials, FIMs). Although the transition region that forms between dissimilar materials has been studied in detail, the influence of co-deposition on the resultant spatial phase distribution and associated mechanical behavior has heretofore not been reported. In this study, FIM samples transitioning from stainless steel (SS) 316 L to Haynes 282 Ni-based superalloy were deposited via directed energy deposition (DED). The FIM samples were compared to baseline, homogeneous single-alloy deposited samples using digital image correlation during tensile testing, together with microscopy, energy-dispersive X-ray spectroscopy, elec-

(DED). The FIM samples were compared to baseline, homogeneous single-alloy deposited samples using digital image correlation during tensile testing, together with microscopy, energy-dispersive X-ray spectroscopy, electron backscattered diffraction, and thermodynamic modeling, to assess the performance of different co-deposition strategies. Each FIM sample exhibited a compositionally and microstructurally unique transition region from SS 316 L to Haynes 282, which was found to have implications on the strain localization across the transition region during uniaxial tensile loading. Finer step sizes in co-deposition were found to minimize strain localization by avoiding sharp compositional interfaces in the transition region.

#### 1. Introduction

Metal additive manufacturing (AM) is a disruptive processing route that has been identified as a driving technology for the most recent industrial revolution: Industry 4.0 [1]. When compared to conventional processing, including casting, forging, and subtractive machining, metal AM has demonstrated a significant advantage in a variety of manufacturing efforts. These include the production of spare parts, prototyping, manufacturing of tools, and part reduction of complex components [2]. A wide variety of metal AM techniques exist that are primarily classified by the form of their feedstock material. Feedstocks include powder, wire, dispersions, as well as filaments. All these techniques rely on the deposition of material on a substrate layer by layer to build a three-dimensional part. To date, powder-based techniques, specifically laser powder bed fusion (LPBF) and directed energy deposition (DED) techniques have achieved the highest levels of technological maturity and industrial use [3].

The features of AM make it possible to design and engineer Functionally Integrated Materials (FIMs) with tunable properties by tailoring microstructure and compositional heterogeneities. This 3D functional design of structural FIMs components is a novel, visionary concept, which will allow the creation of single-structure yet multi-functional products that avoid the need for machining, joining, assembly, coatings, and maybe even wire interconnects. In the ideal case, complex products such as armor with embedded sensors and energy systems could be created in-situ in one machine, requiring only microstructural or compositional variations on an extremely localized level. This revolutionary concept is currently constrained, however, by the lack of fundamental scientific and engineering level understanding of AM, especially when heterogeneities are introduced, which are essential when creating FIMs.

Among AM techniques, DED provides a unique advantage over LPBF in its ability to co-deposit multiple powder feedstocks during the production of a component. This co-deposition is a critical aspect of metal AM that enables the development of functionally integrated materials (FIMs). Conventional materials processing techniques are generally used to achieve homogeneous microstructures that lead to uniform behavior. In contrast, the FIMs concept allows for the three-dimensional functional

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design of structural components through spatial control of materials composition and microstructure to achieve strategically controlled performance in select regions of a fabricated component. Various reviews of the field have articulated the current benefits and challenges of the FIMs concept [4–6]. The development of FIMs via metal AM provides enhanced control to selectively design properties such as strength, corrosion resistance, and operating temperature while minimizing weight and cost [4–6]. Currently, the primary focus of most FIM research is on the development of methodologies to effectively transition between dissimilar material systems within a component without causing material failure.

Most studies in the field of FIMs experimentally form graded transitions between materials [7-13]. Many of these systems transition rapidly from one alloy to another, as demonstrated by L. Bobbio et al. who deposited a block of Ti-6Al-4 V on Invar with a single transition [10]. Due to the additional stable phases and the variability of the liquidus temperature along the compositional transition, sequential deposition of such dissimilar alloys lead to detrimental cracking and delamination in the build near the transition region. Other systems include metal matrix composites (MMCs), as explored by D. Gu et al. in graded depositions of Inconel 718 with TiC particles [12]. Other transitions investigate the application of AM as a replacement for coating techniques, such as the work of M. Ostolaza et al., in which a single layer of H13 tool steel was deposited on top of SS 316 L. The result of no co-deposition of the two powder feedstocks led to a weak metallurgical bond between the coating the base material. The avoidance of deleterious phases along compositional transitions and process development to eliminate cracking and delamination are paramount to the development of FIMs.

FIMs composed of austenitic stainless steels (SS) and Ni-based superalloys have been developed in a variety of forms and exhibit relatively promising printability in transitions from one material to the other [8,13-19]. The systems are mainly based on SS 316 L or SS 304 L transitioning to Inconel 625 or 718. Applications of this FIM system include fields requiring high temperature strength and corrosion resistance such as transportation and energy [19]. The high printability of this system is due to the similarity in the austenitic matrix phase each alloy possesses as well as the high solubility of the primary alloying elements Fe, Ni, Cr, and Mo. While these AM builds generally exhibit good geometric retention when transitioning form SS to Ni-based superalloys, microscopic cracking is observed in several studies. Upon investigation, this cracking is attributed to the known micro-segregation of Nb when depositing Inconel alloys via metal AM, which promotes the precipitation of metal carbide phases, such as NbC, that locally embrittle the material [15]. Some studies have considered the co-deposition strategy for transitioning from SS to Ni-based super alloys. U. Savitha et al. compared a discrete interface between the two materials created by sequential deposition of feedstocks to an interface formed by systematically varying the co-deposition of feedstocks to transition from one to the other [17]. From the successfully deposited samples, tensile coupons were prepared, and stress-strain behavior of the co-deposited samples were compared to the baseline behavior of the two alloys studied. But this global assessment of mechanical behavior is convoluted by the presence of non-uniform composition and microstructure through the tensile coupons gauge section. Y. Su et al. produced several transitions with variable step size in co-deposition of the two materials and found significant precipitation of laves phase due to Nb micro-segregation [16].

To address the challenges of investigating the specific role of step size in the co-deposition during DED, FIM samples were synthesized by codepositing SS 316 L and Ni-based superalloy Haynes 282 with varied step sizes. Haynes 282 does not contain Nb and was selected to avoid the deleterious effects of Nb and carbide formation demonstrated in Inconel systems. In addition, Haynes 282 possesses higher creep resistance and an elevated maximum service temperature when compared to Inconel 718 [20,21]. Systematic characterization, including microscopy and energy-dispersive X-Ray spectroscopy (EDS), of the transitions formed in each FIM sample is used to explain the local strain evolution measured by tensile testing FIM samples with 2D digital image correlation (DIC).

## 2. Experimental procedures

Prior to DED deposition, CALculated PHAse Diagram (CALPHAD) thermodynamic calculations were performed using the ThermoCalc 2022b software. Three databases were considered in this study: the Febased alloy database, Ni-based alloy database, and high entropy alloy database. Each database produced somewhat different predictions, due to the difference in datasets. Upon validation of initial calculations, the Ni-based alloy database was selected for all calculations. This is rationalized by the fact that Ni maintains a concentration above 10 wt% throughout the compositional range of interest, including in the SS 316 L, which satisfies the principal element construct of the Ni-based alloy database. Analysis of the strengthening behavior was also conducted using ThermoCalc Software, by taking advantage of the property module to calculate theoretical yield strength values for any given composition [22]. While this model may account for precipitation, solid solution and grain size strengthening, for this analysis only solid solution strengthening was considered. For the calculations in this study, the matrix phase was selected as FCC L12, the set temperature was 1100 °C, and the strength temperature was set to ambient room temperature (25 °C).

Powder feedstock of SS 316 L and Havnes 282 were purchased from Carpenter Powder Products (Bridgeville, PA) and Praxair, respectively. Both powders were gas atomized in Ar. The nominal chemical compositions of each feedstock powder are shown in Table 1. The DED samples were deposited with a laser-engineering net shaping, LENS®, 750 system (Optomec, Inc. Albuquerque, NM). The LENS® possesses a continuous wave (CW) 1000 W IPG fiber laser (1067 nm), a four-nozzle coaxial powder feed system with two independent powder feeders, a controlled Ar environment glove box (below 20 ppm O), and a motion control system. During deposition, the powder is delivered via a transport gas stream through nozzles that converge at the same point on the focused laser beam to form a melt pool, and a three-dimensional part can be generated line by line and layer by layer via additive processing. Powder feed rate (PFR) calibrations were carried out by measuring the mass of powder delivered through the nozzles over time while varying the motor speed of the powder feeder and the feedstock material. For a given powder feeder, parameter set, and feedstock material, the flow of powder through the powder delivery system was measured for a fiveminute period by recording the mass collected on a scale below the nozzles every five seconds. Each test was repeated five times to assess reproducibility. From these results, polynomial fit equations were used to determine necessary motor speeds to achieve a specific volume percent of powder feedstock at a given layer.

With the powder feedstocks calibrated in the LENS® system, five unique samples were deposited with variable co-deposition conditions describe below. All samples were deposited in a square post geometry (9.65 mm square, 50.8 mm tall) on 6.35 mm thick SS 316 L substrates. Apart from PFRs of the independent powder feeders, all DED processing parameters were kept constant between the samples. The laser power was set to 400 W (corresponding to a measured 280 W at the melt pool) and the laser scan speed was 17 mm/s. Each layer height was 0.25 mm with a single contour. The hatch spacing in a layer was set to 0.41 mm and the hatch angle used was 90° between subsequent layers. The combined global PFR from both powder feeders was maintained at 24 g/ min. Two baseline, homogeneous single-alloy samples were prepared by depositing only SS316L or Haynes 282 during every layer of the build. Three FIM samples were deposited with varying co-deposition step sizes of the co-deposition during transition from SS 316 L at the base to Haynes 282 at the top of each build. Fig. 1a contains a schematic of these FIM samples with black regions representing 100 vol% SS 316 L, white regions representing 100 vol% Haynes 282, and grayscale regions representing co-deposition of both materials. The finest co-deposition step

#### Table 1

Chemical composition of each feedstock powder.

Composition [wt%]	Fe	Ni	Cr	Мо	Mn	Si	Со	Ti	Al	С	Р	S
SS 316 L	Balance	10.3	16.3	2.09	1.31	0.49	N/A	N/A	N/A	0.026	0.026	0.006
Haynes 282	1.5	Balance	20	8.5	0.3	0.15	10	2.1	1.5	0.06	N/A	N/A



Fig. 1. (a) Schematic of the DED build strategies for SS 316 L to Haynes 282 FIMs samples with: (i) a 10% co-deposition step size, (ii) a 50% co-deposition step size, and (iii) no co-deposition; with overlay of tensile coupon orientation and cross section used for microstructural characterization; (b) face on view of the as-deposited FIM sample with 10% co-deposition step size; (c) face on view of the as-deposited FIM sample with 50% co-deposition step size; and (d) face on view of the as-deposited FIM sample with no co-deposition.

size (Fig. 1a(i)) is 10 vol% with 78 layers of 100 vol% SS316L, followed by 45 layers of co-deposition where the vol% of Haynes 282 was increased by 10% every five layers (nine distinct co-deposition regions), and finally 77 layers of 100 vol% Haynes 282. A coarse co-deposition step size (Fig. 1a(ii)) of 50 vol% was deposited with 78 layers of 100 vol% SS316L, followed by 45 layers of co-deposition of 50 vol% SS 316 L and 50 vol% Haynes 282, and finally 77 layers of 100 vol% Haynes 282. The last FIM build ((Fig. 1a(iii))) has no co-deposition of the two feedstock materials with a discrete interface between 100 layers deposited with 100 vol% SS 316 L followed by 100 layers of 100 vol% Haynes 282. Photographs of the resulting deposits are provided in Fig. 1b-c.

All samples were sectioned using electrical discharge machining. Fig. 1a overlays the tensile coupon geometry used. The gauge section of the prepared tensile coupons was 6mm wide and 21.3 mm long with a square cross section. The gauge section was aligned in the co-deposited samples such that the lower quarter of the gauge was composed entirely of SS 316L, the upper quarter of the gauge was composed entirely of Haynes 282, and the central portion of the gauge aligned with the codeposition layers. For the sample with no co-deposition, the gauge section is centered on the discrete interface between the SS 316L and Haynes 282 layers. Baseline, homogeneous single-alloy samples of SS 316 L and Haynes 282 each gauge section exhibits the same position in the build. Each sample had three tensile coupons machined as well as a cross section of the build located adjacent to the gauge section highlighted in Fig. 1a. used for microstructural characterization.

Metallographic preparation of each sample was done through mechanical grinding and polishing procedures. Scanning electron microscopy (SEM) was conducted using an FEI Quanta 3D field emission gun SEM equipped with an Oxford energy dispersive X-ray spectrometer (EDS). EDS maps of local, layer by layer, variation in composition and quantified point measurements across the entire transition region are reported. Electron backscattered diffraction (EBSD) was performed on the 316 L stainless steel and Haynes 282 base alloys using a Tescan GAIA3 scanning electron microscope equipped with an Oxford AztecHKL NordlysMax2 EBSD system. EBSD maps were taken with a step size of 5  $\mu$ m over an area of 3 mm  $\times$  3 mm. The indexed phase in the SS 316 L samples corresponds to an austenitic FCC phase, with a lattice parameter of 3.66 Å. The indexed phase in the Haynes 282 samples corresponds to an austenitic FCC phase, with a lattice parameter of 3.57 Å. The data was post-analyzed using the Oxford HKL Channel 5 software. Optical microscope was performed using an Olympus DSX10-UZH digital microscope. Density calculations were made by thresholding optical micrographs of the sample cross sections and determining the relative area of pores.

To assess the spatial variation in mechanical behavior between the five samples, tensile tests with digital image correlation (DIC) and Vickers microhardness mapping were conducted. An Instron 8800 servo electric frame with a 100 kN load cell was used for testing and the tensile tests were conducted with a quasi-static 0.01 s<sup>-1</sup> strain rate. A thirdparty DIC system from Correlated Solutions was implemented to capture DIC images and the images were captured every 200 ms. The samples were first spray painted with a flat-white paint coat background, and a flat-black speckle pattern was then spray painted on top. The black speckle pattern was meshed and tracked using Correlated Solutions' Vic3D software to create the strain map. Vickers microhardness was measured using a Wilson VH3300 auto-indenter. A grid of indentations was made on the microstructural specimen using a 500 g load, 10 s dwell time, and a 0.37 mm spacing. Rows of nine indents were made along the build direction to produce average values at each layer. Vickers diagonals were automatically measured in the DiaMet software and manually validated in post processing.

## 3. Results and discussion

To determine the role of co-deposition in the resulting properties of FIM components, the compositional range was first explored using CALPHAD calculations of phase stability. The powder feedstock was characterized and calibrated for PFR within the LENS® system. Baseline single-alloy samples of SS 316 L and Haynes 282 were deposited to assess the as-deposited microstructure and mechanical behavior. Finally, FIM samples transitioning from SS 316 L to Haynes 282 were deposited: the first with no co-deposition of the two materials, the second with a transition region possessing a 50 vol% co-deposition step size, and the final with a 10 vol% co-deposition step size. The microstructure and mechanical behavior of each FIM sample was assessed to determine the effectiveness of varied co-deposition strategies in the formation of FIM components.

## 3.1. CALPHAD assessment of FIM transition

CALPHAD calculations served as the basis to predict the phase stability exhibited by the baseline single-phase materials as well as the compositional transitions in the FIMs samples. Fig. 2a and b present equilibrium step diagrams for the composition of the SS 316 L and Haynes 282 powder feedstock, respectively. The calculation is plotted

by the volume fraction of all phases as a function of temperature. It is important to note that these calculations are based on equilibrium thermodynamics of the system and that the actual solidification behavior of the alloys during DED will be kinetically affected by the cooling rate achieved. Nevertheless, this equilibrium phase stability provides valuable insight into the solid-liquid transition as well as the existence of secondary phases. The liquidus temperature is determined from these plots as 1461 °C for SS 316 L and 1369 °C for Havnes 282, which, notably, is 92 °C lower. This difference in melting point is expected to have implications on the processibility, which is further discussed in Section 3.3. Both alloy compositions exhibit a wide solution range for the primary austenitic face centered cubic (FCC)  $\gamma$  phase. SS 316 L has both high and low temperature stability of a body centered cubic phase corresponding to delta and alpha ferrite, respectively. While studies of SS 316 L produced by AM confirm the primary austenitic phase, minor amounts of delta ferrite have been observed [23]. The only other secondary phase predicted for SS 316 L is the sigma phase with a solvus temperature of 866 °C. The sigma phase is a common intermetallic that only forms through long term exposures to temperature, due to the relatively sluggish kinetics of precipitation [24]. Given this low driving force for formation, the sigma phase is not considered detrimental to the as-deposited FIM samples. Haynes 282 also shows stability of a sigma phase with a solvus temperature of 969 °C. The primary



Fig. 2. CALPHAD predictions of phase stability: (a) equilibrium step diagram for the composition of SS 316 L; (b) equilibrium step diagram for the composition of Haynes 282; (c) ThermoCalc material-to-material plot of SS 316 L to Haynes 282 at 750 °C; (d) material-to-material plot of SS 316 L to Haynes 282 at 1050 °C.

precipitate phase in Haynes 282 is the ordered  $L1_2$  FCC phase ( $\gamma$ ) with a solvus temperature of 1012 °C. This phase stability is in good agreement with the known phase formation in Haynes 282 except for the solvus temperature of  $\gamma'$ , which is calculated to be 15 °C higher than what has been experimentally determined [25]. To assess the transition in phase stability from SS 316 L to Haynes 282, material to material calculations were carried out at 750 °C and 1050 °C, Fig. 2c and d, respectively. By fixing temperature in the CALPHAD calculation, volume fractions of phases can be predicted as the composition of the system transitions from 100% SS 316 L, at 0.0 mass fraction of Haynes 282, to 100% Haynes 282, at 1.0 mass fraction of Haynes 282. At the intermediate temperature of 750 °C, the sigma phase stability is generally constant across the compositional transition and the stability of  $\gamma'$  increases with increasing mass fraction of Haynes 282. At the elevated temperature of 1050 °C, a solution window is maintained between the two compositions due to the stability of the primary austenitic FCC phase. From this analysis, no detrimental phase formation is predicted via equilibrium CALPHAD calculations, in agreement with previous AM studies of the alloys, but variations in melting point through the compositional transition may influence processability.

## 3.2. Powder feedstock and powder delivery calibration

The two powder feedstocks were sieved and characterized to determine the contributing factors controlling powder flow and specifically PFR during DED. Fig. 3a presents the powder size distribution (PSD) for SS 316 L and Fig. 3b is a secondary electron (SE) SEM micrograph of the SS 316 L powder morphology. Calculated from the PSD of the SS 316 L

powder, the mean particle diameter was 78 µm with a standard deviation of 20  $\mu m$  and a median D50 particle size of 76  $\mu m.$  The SS 316 L powder morphology can generally be characterized as spherical with some elongated particles. The surface of the powder is smooth, as expected from gas atomization, with attached satellite particles present on most particles. The Havnes 282 powder feedstock has similar characteristics to the SS 316 L powder feedstock. Fig. 3c presents PSD for Havnes 282 and Fig. 3d an SE SEM micrograph of the powder morphology. The mean diameter was 75  $\mu m$  with a standard deviation of 18  $\mu m$  and a median D50 particle size of 79  $\mu m.$  The morphology is spherical with incrementally less large, elongated particles present in the SS 316 L powder feedstock. The surface roughness is very similar and attached satellite particles are observed. From the analysis of the powder feedstocks, the two materials possess considerable similarity in their size, morphology, and PSD.

Fig. 4 illustrates the results of the PFR experiments described in Section 2. The calculated PFR for motor speeds of 1.25-4 rpm are presented for each powder feedstock delivered through powder feeder 1, Fig. 4a, and powder feeder 2, Fig. 4b. The standard deviation error bars for the average PFR demonstrate the relatively minor variation in PFR over the five tests used to produce the plotted average values. Each set of PFR values, given a material and powder feeder, was fit with a polynomial. This provided the best fit which is needed to interpolate the motor speed / PFR relationship at values between the tested increments. The fit equation is critical for planning the deposition of FIM parts to translate the process parameter of motor speed to the resulting volume fraction of each powder feedstock delivered to the molten pool during deposition. The PFR values for the two materials in the two powder





50 µm



Fig. 3. Powder feedstock characterization: (a) particle size distribution plotted as both size-frequency distribution and cumulative sum for the SS 316 L feedstock powder; (b) SEM secondary electron micrograph of SS 316 L feedstock powder; (c) particle size distribution plotted as both size-frequency distribution and cumulative sum for the Haynes 282 feedstock powder; (d) SEM secondary electron micrograph of Haynes 282 feedstock powder.

100



Fig. 4. Average powder feed rate (PFR) observed in the LENS® system at various powder feeder motor speeds for SS 316 L and Haynes 282 for: (a) powder feeder 1 and (b) powder feeder 2.

feeders are similar at motor speed values below 3 rpm. At higher motor speed values, the PFR values for the two powder feedstocks begin to deviate, more so in powder feeder 1. In both powder feeders at higher motor speed values, SS 316 L exhibits lower PFR than Haynes 282. When considering that the nominal densities of SS 316 L and Haynes 282 are 7.99 g/cm<sup>3</sup>, and 8.22 g/cm<sup>3</sup>, respectively, it is expected that the density of the powder, given similar size and morphology, affect the overall dynamics of each feedstock powder flow through the powder delivery system.

#### 3.3. Baseline single-alloy deposited samples of SS 316 L and Haynes 282

The microstructures of the 100% SS 316 L and 100% Haynes 282 single-alloy deposits were characterized, and their mechanical behavior assessed for comparison purposes. Fig. 5 shows the microstructure of the SS 316 L sample (Fig. 5a) and the Haynes 282 sample (Fig. 5b) along the build direction in the gauge section. The final polish used to prepare the specimen led to a partial etching that reveals grain structure in the optical micrographs. A columnar grain structure is observed within both baseline samples. This structure was verified using EBSD as seen in the insets of Fig. 5, where high angle grain boundaries (above 15°) are colored in black and low angle boundaries (2–15°) are colored in grey.

The two materials possess a single austenitic  $\gamma$  phase in the as-deposited state. Both baseline samples indexed well to a single-phase FCC crystal structure throughout the gauge sections, with a lattice parameter of 3.66 Å for SS 316 L and a lattice parameter of 3.57 Å for Haynes 282. This single-phase state agrees well with the CALPHAD predictions discussed above as well as previous studies of the AM microstructures of these alloys [20,26]. Assessment of the grain size from EBSD analysis reveals no appreciable change in the grain size through the gauge section. For the SS 316 L, the higher and lower build height EBSD maps yielded grain size measurements of  $117.6\pm70.5\,\mu m$  and 117.1 $\pm$  72.6 µm, respectively. For the Haynes 282, the higher and lower build height EBSD maps yielded grain size measurements of 159.5  $\pm$  236.6 µm and 169.4  $\pm$  258.5 µm, respectively. This uniform grain structure for each material across the gauge section suggests that the material did not experience sufficiently high thermal energy accumulation during deposition, also referred to as heat accumulation, to cause significant grain growth. There is, however, a significant difference in grain structure when comparing the SS 316 L and Haynes 282 baseline samples as indicated by the significantly higher standard deviation in grain size values for the Haynes 282 baseline sample. Both exhibit columnar grains with orientations relating to the layer scan direction, as previously reported [26], but the epitaxial orientation relationship



Fig. 5. Optical micrographs, EBSD orientation and phase maps of the DED-deposited baseline homogeneous single-alloy samples of: (a) SS 316 L and (b) Haynes 282.

between layers in the Haynes 282 persists far longer through subsequent layers than in SS 316 L, resulting in a larger overall grain size. The most pronounced difference between the SS 316 L and Haynes 282 baseline samples is the significant spherical porosity present throughout the gauge section in the Haynes 282 microstructure. The calculated relative density of the SS 316 L baseline sample is 99.9% dense while the Haynes 282 baseline sample has a relative density of 98.1%. In addition to the porosity, the layer thickness of the Haynes 282 microstructure becomes irregular with increasing height along the build direction. The variation in defect concentration between the two materials is attributable to the fixed DED parameters used to deposit each sample, which were optimized for the deposition of SS 316 L [27]. As reported in Section 3.1, the liquidus temperature of SS 316 L composition is 92 °C above that of Haynes 282, meaning for the same energy input from the laser, the Haynes 282 will melt more readily. During DED, the viscosity and surface tension of a given material have been established as controlling factors of the molten pool [28]. To compare these thermophysical properties between SS 316 L and Haynes 282, additional CALPHAD calculations were performed at 1600 °C, where both alloys would be entirely molten. The dynamic viscosity predicted for SS 316 L is 5.5 mPa's and for Havnes 282 is 4.7 mPa's. These predicted values are on the order of magnitude of previously measured viscosities of molten Fe and Ni [29,30]. Like the predicted viscosity, the surface tension is calculated to be lower for Haynes 282 than SS 316 L, at 1.6 J/m<sup>2</sup> and 1.8 J/m<sup>2</sup>, respectively. It is surmised that the combined lower melting temperature, viscosity, and surface tension of the Haynes 282 alloy are driving factors for the increased defect density and loss of layer-by-layer dimensionality in the DED baseline samples.

Mechanical tensile tests with strain mapping via DIC were performed to determine the mechanical behavior of the baseline single-alloy samples. Fig. 6a contains representative engineering stress-strain curves for the baseline SS316L and Haynes 282 samples. The average mechanical properties from three tensile tests are reported in Table 2. These average

#### Table 2

Mechanical properties of the baseline homogeneous single-alloy SS 316 L and Haynes 282 DED samples measured from the DIC tensile test data using global strain measured across the entire gauge length.

Build	Yield Strength	Ultimate Tensile Strength	Strain-at-
	(YS)	(UTS)	failure
SS 316 L Haynes 282	$\begin{array}{c} 338 \pm 1 \text{ MPa} \\ 689 \pm 27 \text{ MPa} \end{array}$	$615 \pm 3$ MPa 964 $\pm 28$ MPa	$\begin{array}{l} 70\% \pm 0.3\% \\ 35\% \pm 10\% \end{array}$

values of yield strength (YS), ultimate tensile strength (UTS), and strainat-failure are in good agreement with previously reported values for asdeposited material [20,23]. While the strength of Haynes 282 is significantly higher than that of SS 316 L, the strength is well below the expected value for Haynes 282 with an optimally precipitated  $\gamma$ - $\gamma'$ microstructure. This is expected due to the rapid solidification that occurs during DED and the relatively sluggish precipitation behavior of  $\gamma'$ in Haynes 282 [31]. In addition to the absence of the strengthening precipitate in Haynes 282, the large columnar microstructure observed in Fig. 5b further contributes to lower-than-expected strength levels as predicted by the Hall-Petch relationship [32]. When comparing the two materials, the standard deviations in mechanical properties are systematically larger for Haynes 282, especially for the strain-at-failure. This correlates well with the larger defect population in Haynes 282, as porosity often serves as a premature failure site in AM samples, as previously reported [33]. Fig. 6b and c present snapshots of the DIC analysis for SS 316 L and Haynes 282, respectively. Each frame corresponds to a specific global strain value highlighted on the engineering stress-strain curves of Fig. 6a. The color map overlayed on the frames shows the value of normal strain along the tensile axis at each position in the gauge section. Prior to necking, which is observed in the final frame for each material, the strain across the gauge section is highly uniform. Uniform strain confirms that a uniform microstructure has been loaded



Fig. 6. Tensile testing results for the DED-deposited baseline homogeneous single-alloy samples of SS 316 L and Haynes 282: (a) engineering stress-strain curves; (b) DIC frames of local axial strain at various levels of global strain for SS 316 L; (c) DIC frames of local axial strain at various levels of global strain for Haynes 282.

throughout the gauge section.

## 3.4. FIMs samples with variable co-deposition step sizes

Microstructural assessment through the transition of each FIM sample was performed to investigate the effective control over composition during deposition and to evaluate the impact of the co-deposition step size on mechanical behavior.

Fig. 7a-c presents the analysis of the FIM sample with no codeposition. Fig. 7b shows an optical micrograph of the gauge section of the build where both the composition and microhardness along the build direction are quantified in Fig. 7a and c, respectively. Fig. 7a shows the measured concentrations of primary alloy elements Fe, Ni, Mo, and Ti as they vary across the build direction. Horizontal error bars demonstrate the variability in composition at each position. In the lower half of the gauge section, the composition is consistent with that of SS 316 L, and the composition of the upper half is consistent with Haynes 282, albeit with a lower-than-expected value for Fe; this is attributed to poor fitting of the EDS spectrum, as Fe and Co exhibit significant overlap. Microstructural features, namely spherical porosity, in the two regions are comparable to the features found in the two-baseline singlealloy samples discussed in Section 3.3. The compositional transition in Fig. 7a confirms the control of each powder feedstock during DED. It is important to note that the position along the build direction where the transition occurs is not precisely at the designed height of 25.4 mm. This is due to the inevitable variation in layer thickness that occurs during deposition [34]. Nevertheless, the experimental position of the transition is consistent between the EDS compositional analysis and microhardness measurement. The single discrete interface achieved between SS 316 L and Haynes 282 shows clear metallurgical bonding in the optical micrograph, while EDS analysis indicates several layers with transitional compositions. EDS mapping of the interface, Fig. 8, reveals these transitional layers that are quantified in Fig. 7a and their relative local concentrations of Fe, Ni, and Mo. Due to the remelting of subsequent layers during DED, the initial layers of 100 vol% Haynes 282 powder feedstock exhibit Fe leaching from the layers below. Moreover, this remelting leads to heterogeneous distributions of alloying elements within individual molten pools, visualized as swirls in the EDS elemental maps of Fe and Ni. This is a known phenomenon in DED, particularly during co-deposition of multiple feedstock materials, which has been remedied through the design of intermediate repass scans of the layer to remelt the material thus promoting chemical homogeneity [35]. Microhardness across the transition of the FIM sample with no co-deposition exhibit two regions with uniform hardness values. The average hardness for the lower SS 316 L region is 170 HV0.5 and the upper Haynes 282 region is 355 HV0.5. At the same build height as the compositional transition is observed, the hardness increases from the SS 316 L value to the value of Haynes 282. From this analysis we can confirm that the interface created has a higher hardness than SS 316 L. From this FIM sample the ability to transition from one material to another is demonstrated.

Fig. 7d-f presents the analysis for the 50 vol% co-deposition step size. As opposed to the FIM sample with no co-deposition, this transition creates three distinct regions in the gauge section. The lower quarter is composed entirely of SS 316 L, and the upper quarter is composed entirely of Haynes 282, as confirmed by the composition and micro-hardness values. The middle half of the gauge section contains the transition region where the two materials are deposited with a 50 vol% step size (i.e., deposited in a one-to-one ratio throughout the region). The compositional analysis confirms an even mix of the SS 316 L and Haynes 282 through this region, most clearly seen in the even steps of the Mo concentration line. The hardness of this transition region is not simply an average of the two materials but is 199 HV0.5, which is closer to that of SS 316 L. Rule of mixtures does not apply to the hardening across this transition region due to the complex variation in multiple solid solution strengthening elements. The transition region has

increased defect concentrations relative to the SS 316 L material, due to the incorporation of Haynes 282 alloy elements and the associated processing defects mentioned in Section 3.3. The interfaces between regions, like the FIM sample with no co-deposition, are distinct in the microstructure and span several layers due to remelting of subsequent layers. This 50 vol% step size FIM sample confirms the ability to control co-deposition at a coarse scale.

Fig. 7g-i contains the analysis of the finest co-deposition step size studied, 10 vol%. As with the previous 50 vol% step size FIM sample, the gauge section contains three distinct regions with the lower and upper quarters entirely composed of SS 316 L and Haynes 282, respectively. The middle half the of the gauge section is composed of the transition region. The compositional analysis shows a gradual and relatively linear transition between the composition of the two materials through this region. The microstructure also exhibits more gradual transitions in features from region to region making the interfaces between the regions more diffuse. This is due to the less abrupt changes in composition from layer to layer in this FIM sample. As defined in the deposition parameters, the vol% of each feedstock material was kept constant over each of the nine five-layer ( $\sim$ 1.25 mm) increments in the transition region. This step is consistent with the minor fluctuations present in the measured compositions along the build direction. The microhardness across this transition region is nonlinear. At the beginning of the transition, the hardness increases to 180 HV0.5 followed by a drop back to 170 HV0.5, after which it increases, with positive curvature, to the base value of Haynes 282. At the point along this transition where the composition is a one-to-one ratio of the two materials, the microhardness is in good agreement with the microhardness of the transition region in the 50 vol % step size FIM sample described above. This 10 vol% step size FIM sample confirms the incremental control over co-deposition that can be achieved during DED.

To better understand the unique hardening behavior through the transition region of the 10 vol% step size FIM sample, CALPHAD based solid solution strengthening calculations were conducted, as described in Section 2. The theoretical yield strength contribution of solid solution strengthening for each composition along the transition region is plotted in Fig. 9, where the nominal composition from Fig. 7g at a specific build height is used to calculate the strengthening contribution. The trend predicted by this model show little agreement with the hardening behavior measured experimentally. The solid solution strengthening contribution at the composition of Haynes 282 far exceeds the measured vield strength found in Table 2. This discrepancy is perhaps not surprising, as solid solution strengthening in the complex compositional range present in these FIMs samples is difficult to model as these compositions deviate from the conventional solute-solvent compositions that are usually assumed when modeling. This issue has been discussed within the context of high entropy alloys [36-38] and will require careful study in the case of FIMs.

The mechanical behavior of each FIM sample was determined through tensile tests, with DIC used to resolve local strain evolution; resulting engineering stress-strain curves are presented in Fig. 10a. It is important to note these stress-strain curves are created from samples with compositionally and microstructurally non-uniform gauge sections, so comparison to the baseline single-alloy samples is convoluted. The FIM sample printed with no co-deposition exhibits similar strength as the baseline SS 316 L sample with roughly half the strain-at-failure. The 50 vol% step size FIM sample has a similar yield strength to the baseline SS 316 L but upon plastic deformation, the sample presents an enhanced work hardening behavior and fails at an incrementally lower strain value. Finally, the 10 vol% step size FIM sample has a global yield strength lower than the other two FIMs samples and similar strain-at-failure as the 50 vol% step size FIM sample.

These global behaviors of each coupon stem from the local strain evolution and strength variation across each FIM sample caused by the unique compositional transitions of each. The DIC frames of the no codeposition sample (Fig. 10b) reveal that the region of the gauge



Fig. 7. Analysis of each transition region in the FIMs deposited samples: (a),(d), and (g) quantified SEM EDS composition measurements across the transition region for the no co-deposition sample, the 50% co-deposition step size sample, and 10% co-deposition step size sample, respectively; (b), (e), and (h) optical micrographs of the no co-deposition sample, the 50% co-deposition step size sample, and 10% co-deposition step size sample, respectively; (c), (f), and (i) microhardness measurements across the transition region for the no co-deposition sample, the 50% co-deposition sample, the 50% co-deposition sample, the 50% co-deposition step size sample, and 10% co-deposition step size sample, respectively; (c), (f), and (i) microhardness measurements across the transition region for the no co-deposition sample, the 50% co-deposition step size sample, and 10% co-deposition step size sample, respectively.



Fig. 8. SEM EDS of the no co-deposition FIMs sample at the transition from SS 316 L to Haynes 282.



**Fig. 9.** Comparison of the hardening behavior measured across the 10 vol% codeposition step size FIM sample (black) and the calculated theoretical yield strength based on solid solution strengthening (yellow).

section composed of Haynes 282 undergoes near zero deformation throughout the tensile test. By tracking the position of the discrete interface between the two materials in this gauge section, denoted by the yellow dotted line overlayed in Fig. 10b, it is found that the interface coincides with the lowest position in the gauge that undergoes near zero plastic strain. Through each frame, plastic strain is increasingly localized to the SS 316 L region of the gauge section, where eventual necking and failure occurs. This non-uniform strain evolution explains the global behavior described above. Since deformation only occurred in the lower half of the gauge section composed of SS 316 L, the yield strength is that of SS 316 L, but with only half the gauge length to accommodate deformation, the strain-at-failure is halved. A similar trend in strain-atfailure is reported for bulk tensile testing of transition regions from SS 316 L to IN 625 with no co-deposition [17]. This behavior that manifests in the stress-strain curve is convoluted by non-uniform elongation within the gauge section, which is verified through the DIC analysis.

In Fig. 10c, the DIC frames of the 50 vol% step size FIM sample show three distinct regions along the gauge section, each with relatively uniform strain evolution prior to necking. As with the no co-deposition sample, the region composed entirely of SS 316 L accommodates most of the deformation during tensile testing and contains the location of failure. While the Haynes 282 upper region possess near-zero plastic strain, the transition region exhibits a constant strain value that is incrementally lower than the strain formed in the SS 316 L. Over the entire test, this transition region accommodates 13% strain within the co-deposition region. The minor hardening observed in this transition region explains the variation in strain accommodation as well as its uniform nature across this portion of the gauge section. The simultaneous deformation of SS 316 L and this incrementally harder co-deposited region contributes to the enhanced work hardening



Fig. 10. Tensile testing results for the FIMs deposited samples: (a) global engineering stress-strain curves using global strain measurements for each FIMs deposited sample; (b) DIC frames of local axial strain at various levels of global strain for the no co-deposition FIMs sample; (c) DIC frames of local axial strain at various levels of global strain for the 50% co-deposition step size FIMs sample; (d) DIC frames of local axial strain at various levels of global strain at various levels of global strain for the 10% co-deposition step size FIMs sample.

behavior seen in the global stress-strain curve.

When comparing the DIC frames for the 10 vol% step size FIM sample, Fig. 10d, the three regions of strain behavior are present, but with diffuse interfaces due to the more gradual transition achieved in this FIM sample. This sample follows the behavior observed in the other FIM samples in that the lower SS 316 L region accommodates most of the deformation while the Haynes 282 region has near-zero plastic strain. But the strain evolution within the transition region is non-uniform. At the lower portion of the transition region, the strain values are similar to those in the neighboring SS 316 L, with the strain gradually decreasing along the build direction towards the Haynes 282 region. Overall, this transition region accommodates 20% strain during the tensile test and does not have clear interfaces in the strain field. In fact, necking and failure occurs within the lower portion of the transition region. The location of failure aligns with the softening observed in microhardness measurements of the gauge section at the lower portion of the transition region. This behavior also rationalizes the lower global yield strength measured for this FIM sample. Previous reports of global tensile behavior for transition regions of SS 316 L to IN 718 possessing 10 vol% step sizes exhibit similar drops in yield and ultimate tensile strength as compared to the base material as well as reduced global strain-at-failure [16]. For the first time, the relative strain concentration across a 10 vol % step size transition region between a stainless steel and Ni-based superalloy is resolved using DIC analysis.

# 4. Conclusions

FIM samples that transitioned from SS 316 L to Haynes 282 Ni-based superalloy were fabricated using co-deposition in DED. Different co-deposition step sizes were used to compare mechanical behavior with baseline single-alloy depositions of SS 316 L and Haynes 282. The following key points are established by the current work:

- 1. With similar powder size, distribution, and morphology, the density of powder feedstocks influences their resulting flowability through the LENS® powder delivery system.
- 2. While the SS 316 L and Haynes 282 exhibit similar phase formation, differences in thermophysical properties including molten viscosity and surface tension along with melting points lead to increased defect formation in Haynes 282 during deposition. This challenge may be addressed by separately optimizing the Haynes deposition parameters and perhaps tuning the parameters accordingly through the transition region.
- Co-deposition of multiple powder feedstocks produce transitions in the DED build that exhibit different mechanical behavior and strain localization.
- 4. The discrete interface formed by transitioning with no co-deposition spans several layers due to the remelting of subsequent layers during DED.
- 5. Finer co-deposition step sizes reduce sharp compositional interfaces, which control strain localization in the resulting component.

## CRediT authorship contribution statement

MacDonald Benjamin E.: Writing – original draft, Visualization, Methodology, Investigation, Formal analysis. Zheng Baolong: Writing – review & editing, Visualization, Methodology, Investigation, Conceptualization. Lavernia Enrique J.: Writing – review & editing, Supervision, Resources, Investigation, Conceptualization. Schoenung Julie: Writing – review & editing, Supervision, Resources, Investigation, and Conceptualization. Valdevit Lorenzo: Writing – review & editing, Supervision, Investigation, Conceptualization. Jiang Sen: Writing – review & editing, Investigation, Conceptualization. Cao Penghui: Writing – review & editing, Supervision, Investigation, Conceptualization. Fields Brandon: Writing – review & editing, Methodology, Investigation. Wang Xin: Investigation, Formal analysis.

## **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.

### **Data Availability**

Data will be made available on request.

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