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EXPLORING GRADIENT PATHWAYS IN HIGH TEMPERATURE, FUNCTIONALLY GRADED ALLOYS

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A new approach aims to fabricate parts with targeted, site-specific properties for a wide range of applications in extreme environments within the aviation, space, and energy sectors.

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Additive manufacturing (AM) provides a tremendous opportunity to synergistically couple materials, design, and manufacturing strategies. One can strive toward fabricating parts with targeted properties by enabling site-specific metal-metal or metal-ceramic compositional transitions. Parts with site-specific composition and hybrid microstructures are relevant in an application space where certain sections of the component encounter considerably higher temperatures than others. For example, one may design composite parts with Nb-base refractory alloys for hotter sections (requiring increased high temperature strength and creep resistance) while opting for relatively low cost and low gamma prime containing IN718 at zones operating below 1400ºF[1-4]. Note that conventional welding and joining approaches are adopted to deal with parts operating at widely varying temperatures.

For example, Fig. 1 illustrates a commercial, bimetallic joined structure for an engine part where the performance requirements of the airfoils (SX material) and disk section are quite different. Even within a turbine disk, the performance requirement for the outer rim of the disk needs material with a larger-grained microstructure that is creep resistant as opposed to the low cycle fatigue (LCF)-limited, fine-grained core of the disk[5].

FIG 1 GOES NEAR HERE

Although conventional joining processes are limited in their ability to create components comprised of multiple dissimilar materials, AM processing provides an approach to circumvent these restrictions through compositionally grading materials. Considerations include:

Feasibility: The approach allows the transition from one terminal alloy composition to another, even when they are nonweldable[4]. In this case, a gradual compositional gradation via an additive route makes such a transition feasible. In some cases, an intermediate third alloy composition may be selected for nonlinear compositional gradient setup, similar to the use of strike coatings during metal plating operations.

Performance benefit: The goal is to eliminate abrupt property transitions at dissimilar interfaces along with alleviating residual stress across large structures. Apart from alleviating coefficient of thermal expansion (CTE)-driven thermal mismatch between different alloy systems, smooth compositional transitions can also exhibit performance benefits for static, dynamic, and high-speed impact property response. In short, the graded concept can help eliminate the weak link in dissimilar welded parts, which is often the weld zone itself.

Gradient design adapted for AM: While monolithic alloy use has been employed successfully, this is primarily due to the high strength and high temperature capabilities of nickel-base superalloys and refractory alloys. Further benefit for structural components may be realized when site-specific chemistry—tailored to desired performance requirements—is employed. Additional functionalities enabled by AM for topological optimization and manufacturing unitized components with complex geometries may be enhanced by coupling advanced designed tools with fabrication of multi-material parts[6].

Compared to conventional casting, the directed energy deposition (DED) additive modality reduces initial investment by not requiring casting molds. In addition, DED offers near-net shape structures, which significantly reduce machining and material waste compared to forging. High-speed printing via DED can also enable printing of large-scale structures at a fast production rate. The goal of this effort is to fabricate unitized parts with the proposed technology, with a reduction in part count to decrease supply chain costs. The unique challenges of additively manufacturing high temperature alloys involve crack sensitivity with regard to as-built process parameters and post-processing treatments. While the thermal gradients and cooling rates in and around the melt pool control aspects of the liquid-to-solid phase transformation, spatially varied thermal cycles may result in residual stresses and distortion in solidified components (e.g., due to subsequent solid-state transformations)[7, 8].

Material properties and deformation characteristics also depend on the actual alloy class being studied. For example, low gamma prime Ni-base superalloys like IN718 and IN625 are readily weldable, and AM build parameters dictate thermal gradients and cooling rate dependencies on microstructure.

However, the thermal gradient effect and cooling rate-dependent transformation kinetics are quite different for Nb-base refractory alloys like C103[9, 10]. Figure 2 is an illustration of varied operating temperatures among different alloy classes[9]. Note that for monolithic builds, the alloy of choice restricts us, either by cost or performance, to operate within a specific temperature range. The proposed compositional transition from a high-temperature Ni-base superalloy (e.g., IN718) to an extreme temperature-capable Nb-base refractory alloy (e.g., C103) will allow fine-tuning of microstructures for zone-based performance over a wider temperature range.

FIG 2 GOES NEAR HERE

In addition, this concept helps to simultaneously investigate the complex phase transformations, deformation mechanisms, and interrelationships across graded alloy chemistries. This effort is relevant to a wide range of applications in aviation (structural engine components), space (access, high velocity) and energy fields (marine, nuclear, and renewables). For example, NASA and Boeing have adopted zone-based deployment of lightweight and high-temperature materials for the rocket nozzles and hypersonic air breathing structures (X-15, X-43A, and X-51A), respectively[11, 12].

APPROACH AND RESULTS

The proposed study of developing a graded structure from IN718 to C103 alloy may be broken down into the following categories: (a) find thermodynamically stable, kinetically feasible gradient pathways between terminal alloys from two different alloy classes—Ni-base superalloy (IN718) used for high temperature applications and Nb-base refractory alloy (C103) capable of operating at extreme temperatures; (b) adopt optimal build strategies to additively manufacture crack-free graded structures with hybrid microstructures; (c) understand phase transformation, microstructural evolution, and variation in residual stress as a function of thermal history (as-built and post-processed states); and (d) correlate these with the mechanical property response of graded specimens. Keep in mind that this is a work in progress and the current manuscript primarily deals with the first task, which is CALPHAD-based analyses of IN718 to C103 graded structures.

Design of the gradient pathway between two terminal alloys is a critical step as it ensures build compatibility, both thermodynamically and kinetically. For this study, Pandat^{TM} (a CALPHAD-based thermodynamic software) was employed to understand the variations in solidus and liquidus temperatures within the two terminal alloys. Along with this, important parameters like gamma prime solvus temperature, equilibrium phase fractions as a function of temperature, and corresponding phase compositions were also determined. Initial trials involved identifying the major equilibrium phases in a pure Ni to pure Nb graded structure (Fig.3).

FIG 3 GOES NEAR HERE

For this calculation, 11 major phases, namely liquid (L), face centered cubic (fcc), body centered cubic (bcc), ordered bcc (B2), gamma prime ($L1_2$), gamma double prime, delta, mu, sigma, Laves, and $AlNbNi_2$, were selected. As shown in Fig. 3, the Ni-Nb binary phase diagram exhibits an fcc crystal structure on the pure Ni side, and a bcc crystal structure on the pure Nb end. There are also intermediate intermetallic phases—delta and mu, with possible compositions of Ni_3Nb and Ni_6Nb_7, respectively, coupled with associated eutectic reactions.

Subsequently, in the modeling effort for commercial IN718 and C103 alloys, a six-zone linear compositional gradation was chosen to predict the phase evolution of these 11 phases. Thus, if the compositional variations may be defined as (x*(Ni alloy) + y*(Nb alloy)), where a and b are weight fractions of the two terminal alloys and x + y = 1, then the gradient steps for six-zone linear compositional gradation were set up such that the unicompositional zones are with x = 1, 0.8, 0.6, 0.4, 0.2, and 0. Thus, the terminal alloy compositions were represented by x = 1 (IN718 with nominal composition of 54Ni-0.7Al-19Cr-17Fe-3Mo-5.3Nb-1Ti wt%) and x = 0 (C103 with nominal composition of 89Nb-10Hf-1Ti wt%).

CALPHAD-based thermodynamic predictions of this compositionally graded structure are compiled in Tables 1-3. Table 1 shows a tabulation of predicted solidus and liquidus temperatures for each unicompositional zone in the six-zone linear compositionally graded model. The table clearly shows a significant variation in freezing range, which is the difference between liquidus and solidus temperatures. This is also an indication of how quickly a liquid solidifies: (i) in a layer-by-layer fashion for materials having smaller freezing ranges (e.g., eutectics), or (ii) for an extended period of time resulting in elongated grains and possibly with enhanced hot tearing tendencies in alloys that have relatively large freezing ranges.

TABLE 1 GOES NEAR HERE

Similar to the CALPHAD results for pure Ni-Nb gradation, these predictions also indicate the formation of fcc and bcc phases for two terminal alloy compositions. However, for intermediate compositions, a C14 Laves phase is predicted as the first phase to solidify. Pandat^{TM} software was also used to predict equilibrium phase fractions at both room (25^{o}C) and elevated (1000^{o}C) temperatures, as compiled in Tables 2 and 3, respectively. These tables indicate that as the composition of a block is altered from IN718 to C103 in a stepwise manner, the primary phase with the highest phase fraction changes from fcc to intermediate delta and C14 Laves phases, and finally to a bcc crystal structure.

TABLES 2 AND 3 GO NEAR HERE

It is evident that these equilibrium thermodynamic predictions point toward considerable challenges—such as hot cracking and distortion due to residual stress—associated with additively building a IN718 to C103 graded structure. However, a blown powder DED modality provides a unique opportunity to regulate the deposition rates of terminal alloys via a multi-hopper powder feed system, along with the potential to regulate the substrate and surrounding temperature. Note that prior studies have discussed the nuances of laser-processed microstructures and defect distribution in Ni-base superalloys[13-16] and Nb-base refractory alloys[17-20]. Based on the above calculations, Fig. 4 shows a schematic of IN718 to C103 graded block that will be used for initial build trials.

FIG 4 GOES NEAR HERE

As indicated in the figure, the build height of two terminal compositions will be approximately 40 mm, while each of the intermediate four layers will be 5 mm tall. The overall block size of 100 x 25 x 10 mm may be subsequently machined to extract specimens for characterization and testing of composite builds. Based on the literature, at least two or three build trials will likely be required to identify overlapping process parameters between the terminal alloys, as well as to conduct post-build analyses to ensure defect-free builds. A successful gradient build via one combination of gradient step size and pathway would be considered a sufficient criterion to move forward with post-build characterization. During this step, thermodynamic predictions could be validated, and heat treatment and mechanical property response could also be investigated.

CONCLUSION

The proposed research effort delves into one of the many unique AM functionalities, by using a combinational approach to generate hybrid microstructures tailored for site-specific property response. This material-agnostic concept is currently being studied on alloys that are used for high temperature and extreme environment applications—namely Ni-base superalloys (IN718) and Nb-base refractory alloys (C103), respectively. Preliminary CALPHAD-based thermodynamic predictions show significant variations in freezing ranges and primary phases to form within the individual unicompositional zones in a six-zone linear compositionally graded model. The equilibrium phase fractions at both room (25^{o}C) and elevated (1000^{o}C) temperatures point toward essentially different solidification and solid-state transformation routes at the intermediate unicompositional steps.

Based on this data, a plan was developed to additively build a graded structure with a defined gradient step size and pathway. Future research includes post-build characterization coupled with a fundamental evaluation of phase transformation and deformation mechanisms in these graded structures. This approach provides a critical path toward a broader vision of employing breakthrough AM functionalities with integrated design, manufacturing, and materials evolution criteria.

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FIGURE CAPTIONS

Fig. 1 – Illustration of site-specific property requirements in an aircraft engine part. The airfoil sees much higher temperature than the blade, hence the former is SX as opposed to PX disks. Within the disks there is a need for zone-based microstructures – LCF-limited core with finer grain size and creep-limited rim with coarser grains[5].

Fig. 2 – Tensile strengths of various alloy classes as a function of temperature[9].

Fig. 3 – Ni-Nb binary phase diagram as predicted by Pandat^{TM} software.

Fig. 4 – Schematic of a six-zone compositionally graded structure with approximate block dimension.

CALLOUTS

FIG 1

Single-crystal airfoils

Polycrystalline turbine disk

Conventional blade and disk assembly

-30% weight

Blisk (bladed disk)

Hybrid turbine disk

Material B:

- Higher toughness
- Better LCF
- Finer grain size

Material A:

- Higher thermal stability and oxidation-resistant properties
- Better creep-resistant properties
- Larger grain size

FIG 2

Ultimate tensile strength, MNm^{-2}

600 400 200 0

Temperature, °C

0 200 400 600 800 1000 1200

Ti alloys Al Ferritic alloys Superalloys Refractory metals

FIG 3

Temperature, °C

2500 2000 1500 1000 500 0

Wt%, Nb

Ni Nb

0 20 40 60 80 100

Ni to Nb – 11 phases

Fcc Fcc + liquid Delta + liquid Liquid Liquid + Mu_Phase Bcc + liquid

Delta + Fcc Delta Delta + Mu_Phase Mu_Phase Bcc + Mu_Phase

FIG 4

Build direction Substrate

Alloy C103 Alloy IN718

1 in. = 25 mm

10 mm

5 mm

4 in. = 100 mm

TABLES

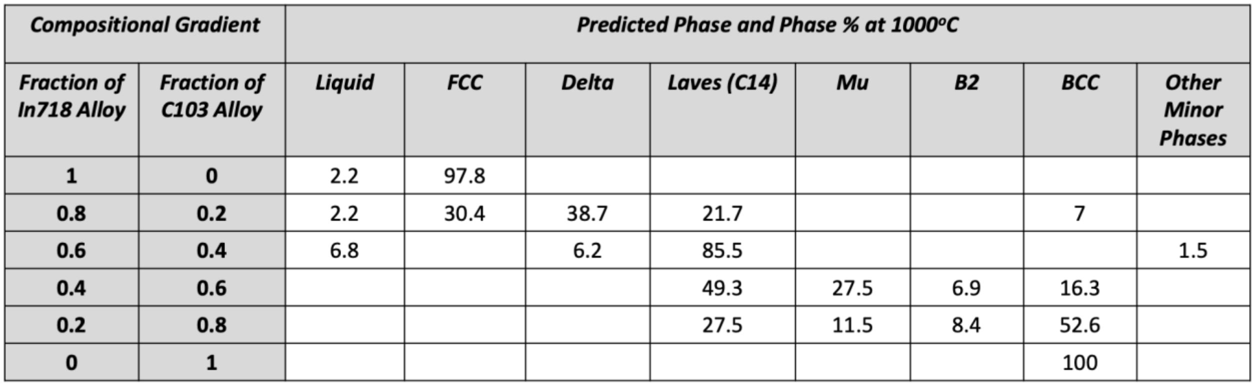
TABLE 1 – PREDICTED SOLIDUS, LIQUIDUS, AND FIRST PHASE TO SOLIDIFY FOR SIX-ZONE LINEAR COMPOSITIONALLY GRADED MODEL

Fraction of In718 Alloy	***Fraction of C103 Alloy***	***Liquidus Temp (°C)***	***Solidus Temp (°C)***	***Freezing Range (Del °C)***	***Primary Solid Phase to form in Liquid***
1	**0**	1330	1260	70	FCC
0.8	**0.2**	1279	939	340	Laves (C14)
0.6	**0.4**	1510	844	666	Laves (C14)
0.4	**0.6**	1533	1015	518	Laves (C14)
0.2	**0.8**	2019	1005	1014	BCC
0	**1**	2411	2372	39	BCC

TABLE 2 – PHASE PREDICTION AND CORRESPONDING PHASE FRACTION AT 25^{O}C FOR SIX-ZONE LINEAR COMPOSITIONALLY GRADED MODEL

Compositional Gradient		***Predicted Phase and Phase % at 25°C***							
Fraction of In718 Alloy	***Fraction of C103 Alloy***	***FCC***	***Gamma Prime $(L1_2)$***	***Delta***	***Laves (C14)***	***Mu***	***$AlNbNi_2$***	***BCC***	***Other Minor Phases***
1	**0**	45	11.6	19.7		5.6		18.1	
0.8	**0.2**			53.7	4.8	5.1	5.2	28	3.2
0.6	**0.4**			26.6	62.2		4.3	2.3	4.6
0.2	**0.8**				27.5	12.5	1.5	52.1	6.4
0	**1**							92.7	7.3

TABLE 3 – PHASE PREDICTION AND CORRESPONDING PHASE FRACTION AT 1000^{O}C FOR SIX-ZONE LINEAR COMPOSITIONALLY GRADED MODEL



Compositional Gradient		***Predicted Phase and Phase % at 1000^{o}C***							
Fraction of In718 Alloy	***Fraction of C103 Alloy***	***Liquid***	***FCC***	***Delta***	***Laves (C14)***	***Mu***	***B2***	***BCC***	***Other Minor Phases***
1	**0**	2.2	97.8						
0.8	**0.2**	2.2	30.4	38.7	21.7			7	
0.6	**0.4**	6.8		6.2	85.5				1.5
0.4	**0.6**				49.3	27.5	6.9	16.3	
0.2	**0.8**				27.5	11.5	8.4	52.6	
0	**1**							100	