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Functionally graded material of 304L stainless steel and inconel 625 fabricated by directed energy deposition: Characterization and thermodynamic modeling

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ABSTRACT

Many engineering applications, particularly in extreme environments, require components with properties that vary with location in the part. Functionally graded materials (FGMs), which possess gradients in properties such as hardness or density, are a potential solution to address these requirements. The laser-based additive manufacturing process of directed energy deposition (DED) can be used to fabricate metallic parts with a gradient in composition by adjusting the volume fraction of metallic powders delivered to the melt pool as a function of position. As this is a fusion process, secondary phases may develop in the gradient zone during solidification that can result in undesirable properties in the part. This work describes experimental and thermodynamic studies of a component built from 304L stainless steel incrementally graded to Inconel 625. The microstructure, chemistry, phase composition, and microhardness as a function of position were characterized by microscopy, energy dispersive spectroscopy, X-ray diffraction, and microindentation. Particles of secondary phases were found in small amounts within cracks in the gradient zone. These were ascertained to consist of transition metal carbides by experimental results and thermodynamic calculations. The study provides a combined experimental and thermodynamic computational modeling approach toward the fabrication and evaluation of a functionally graded material made by DED additive manufacturing.

1. Introduction

Functionally graded materials (FGM) are highly desirable in applications in which the service conditions of a part vary with location and therefore the material requirements also vary with location [1]. Extreme-environment applications such as in aerospace [2] or nuclear power generation require parts that, for instance, must perform at radically different temperatures at different locations in the part [3]. Therefore, such properties as corrosion and oxidation resistance, strength, toughness, wear resistance, light weight, and reasonable cost may all be required in, for instance, various regions of an engine component, but are rarely if ever all found in a single material [4,5]. Joining of dissimilar materials or functional grading of two or more materials is therefore required [1,6]. Functionally graded materials are divided into two types: those made by construction, such as adding layers to build up a part, and those made solely by mass transport, or use of diffusion to create a gradient [2].

Conventional fusion welding is routinely used to join similar metals, and sometimes to join dissimilar metals. However, it is common when welding dissimilar metals to use intermediate layers that separate the two dissimilar metals [7], or to use high-energy-density techniques such as laser welding to prevent such problems as cracking or embrittlement [8]. These issues are usually caused by lack of solubility, atomic structure mismatch, variation in thermal expansion, and the formation of thermodynamically stable brittle intermetallic phases along the gradient composition [8]. Irregular compositional inhomogeneities in the weld also present difficulties such as susceptibility to corrosion and hydrogen cracking [9].

Avoiding melting altogether through the use of solid state joining processes (e.g. ultrasonic welding (UW) and friction stir

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welding (FSW)) is one way to prevent cracking or embrittlement in the joining of two dissimilar metals. In FSW, the two workpieces are severely deformed by a much harder tool and the weld is formed in the solid state [10]. This develops a purely mechanical bond, avoiding the above-mentioned issues that can occur in fusion welding, but creates a very narrow boundary zone between the two metals rather than a gradient of composition [11]. However, differences in material properties between the two component materials can result in thermal and mechanical residual stresses in a structural component. Use of a graded structure without a narrow boundary zone can reduce the occurrence of these stresses [2,12].

The FSW technique can also be applied to create bulk materials with a gradient of properties through control of grain size with the possibility of creating gradient microstructures. Instead of welding, friction stir processing (FSP) and submerged friction stir processing (SFSP) can be used to change the properties of a metal in bulk by applying severe plastic deformation over a larger area [13].

Additive manufacturing (AM) is ideally suited to the constructive method of preparing functionally graded materials. In particular, the directed energy deposition (DED) method, in which powder is fed into the melt pool under a moving laser [14], can be employed for FGM by varying the powder composition between layers, which is readily done using a deposition machine equipped with two or more powder feeders [15,16]. This process has been used to prepare graded structures of titanium alloys [15–18]. However, as DED is a fusion-based process, the potential problems of dissimilar metal welding, namely, the formation of undesirable intermetallic phases, must be addressed [11,19].

The nickel-based superalloy Inconel 625 (IN625) and grade 304L stainless steel (SS304L) were selected for this study. These alloys both exhibit a single face-centered cubic crystal structure from the melt down to room temperature, with no allotropic phase transformations, and the principal constituents of the alloys, namely Fe, Cr, and Ni, have good solubility. As a graded alloy system, it would be useful in applications that require both strength and corrosion resistance at elevated temperature, where IN625 is suitable, and lower cost and mass, where SS304L is suitable. One such application is in high-end automobile engine valve stems [11] while another application is in functionally graded, corrosion resistant coatings. The fabrication of both SS304L and IN625 parts by additive manufacturing has been studied [20–23]. Furthermore, gradients of nickel-based alloys to stainless steels have been previously studied [24–26]. These have consisted of 316 stainless steel graded to a precipitation-hardenable nickel-based alloy such as Inconel 718 [24] or Rene88DT [25,26]. However, when these alloys are fabricated using additive manufacturing, the microstructure is effectively in the solution-treated condition [27], and subsequent heat treatments are required to develop the mechanical properties of wrought material [28]. Therefore, the non-precipitation-hardenable alloy IN625 was selected for this study.

Selective laser melting (SLM), a variant of additive manufacturing in which powder is spread in layers and locally melted with a laser heat source, has been used to fabricate SS304L [20,21]. The microstructure of these parts is principally austenitic and features appear that correspond to individual laser passes. Directed energy deposition has also been used to create parts of austenitic stainless steel [29–31]. These studies, which have focused mainly on process optimization, have succeeded in producing parts with higher yield and ultimate strengths than wrought material, although with reduced ductility.

In IN625 deposited by directed energy deposition, the microstructure shows dendrites oriented parallel to the build direction and which appear as cellular when viewed perpendicular to the build direction [22]. These observations are similar to those reported by Dinda and co-workers [23], who observed that dendritic structures follow the direction of heat flow.

In this work, a part fabricated by DED with a structure graded from type SS304L to the nickel-based alloy IN625 was evaluated for elemental composition, microstructure, phase composition, and mechanical properties. The structure and properties of the gradient zone were compared to the constituent alloys deposited by DED at each end of the process. The phases found in the gradient zone were compared to those obtained by CALculation of PHAse Diagrams (CALPHAD) modeling to ascertain the viability of using CALPHAD to predict the appearance of the experimentally observed structures in this system.

2. Experimental

A square post, 16 mm wide and 34 mm tall, was built on a substrate of SS304L as shown in Fig. 1. IN625 and SS304L powders of compositions noted in Table 1 were used (Carpenter Technology Corporation). The gradient part was built using a directed energy deposition system (RPM 557 Laser Deposition System) under an argon atmosphere. This system allows up to four powders to be added to the build during fabrication and the volumetric fraction of each powder can be changed by about 1% per deposited layer. Twenty layers of SS304L were deposited before grading began. In the graded region, the volume of SS304L powder was reduced by 4% and IN625 powder increased by the same amount in each successive layer, for a total of 24 layers. Nineteen layers of IN625 were deposited on top of the gradient zone. Layers were approximately 0.5 mm tall and were built by a 910W YAG laser with a hatch angle of 60°.

The post was sectioned using wire electric discharge machining as shown in Fig. 1. The cut section was mounted in epoxy and prepared using standard metallographic techniques. The sample was etched electrolytically in 10% oxalic acid for 10 s at 4 V. The microstructure was evaluated using visible-light microscopy (Keyence VHX).

X-ray diffraction patterns were collected using a Bragg-Brentano-type diffractometer (Panalytical Empyrean) with a θ–θ goniometer. The X-ray source was a CuKα tube operated at 45 kV and 40 mA and having a wavelength of 1.54 Å. Variable incident and diffracted-beam slits were used to obtain a line focus of 10 mm by 1 mm, allowing a narrow range of composition in the gradient zone to be analyzed. An area detector (Pixcel 2.0) in 1D scan mode was used to collect the diffracted beam. As variable-slit diffraction patterns inherently possess a sloped background, the patterns were converted to fixed-slit equivalents in postprocessing (Jade 2010).

Energy dispersive spectroscopy (EDS) was performed in a scanning electron microscope (FEI Quanta 200) with a silicon drift detector (Oxford X-act PentaFET Precision). The spectra were collected and analyzed in a semi-quantitative manner (Oxford AZtec version 2.4). It should be noted that all analyzed compositions are in weight percent. The planned composition, which was varied volumetrically, was converted to planned weight percent using the densities of IN625 and SS304L.

Microhardness was measured using Vickers indenter (Leco V-100-C1) with a load of 300 g and a dwell time of 15 s. A minimum of five indents were taken for each layer of the sample analyzed.

3. Computation

Because the mechanical properties of alloys are strongly coupled to the relative stability of constituent phases, which gives rise to particular morphologies at the micro-scale, thermodynamic analyses of these systems yield insight into the propensity of gradient alloys to precipitate undesirable phases during processing. The equilibrium phase diagram serves as a first-order indication for this