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Thermal cycling of Fe_3Al based iron aluminide during the wire-arc additive manufacturing process: An in-situ neutron diffraction study

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ABSTRACT

Fe_3Al based iron aluminide has continuously been attractive because of its excellent oxidation resistance, corrosion resistance, light weight and low material cost. It has been considered as a promising replacement of regular stainless steel in fossil energy industry. However, the industrial application of iron aluminide is limited by its low room temperature ductility and high fabrication cost. In recent years, additive manufacturing processes have been proved capable of producing iron aluminide with relatively lower cost as compared to traditional powder metallurgy processing. In the present research, the influence of thermal cycling during the additive manufacturing of Fe_3Al based iron aluminide on the phase fraction inside the deposited material has been simulated and investigated using in-situ neutron diffraction. Upon heating, the Fe_3Al based iron aluminide has experienced $Fe_3Al \leftrightarrow FeAl$ phase transformations, FeAl phase ordering-disordering, and Fe_3Al phase transformation from imperfectly ordered B2 structured to perfectly ordered $D0_3$ structure. Also, the existence of the forbidden Fe_3Al 110 reflection has been determined by neutron diffraction and further evaluated. In addition, the variation of phase fractions throughout the heat treatment has been quantitatively analyzed by Rietveld refinement.

1. Introduction

Fe_3Al based iron aluminide has been considered as a promising replacement of regular stainless steel in piping and tubing for fossil energy systems, due to a combination of advantages such as excellent oxidation and sulfidation resistance, considerable high temperature strength and creep resistance, low density and low cost [1]. Extensive efforts have been made to improve the room-temperature ductility and high-temperature strength of iron aluminide, by means of adding alloying elements and heat treatments [2]. To date, the room-temperature tensile elongation of Fe_3Al based iron aluminide has been improved up to 11% with the selection of suitable alloying elements and appropriate thermo-mechanical processing combined with subsequent annealing [3]. On the other hand, these combined processing techniques have on the other hand increased the manufacturing cost of this alloy and counterbalance their advantages.

For the successful introduction to the market, a cost-effective manufacturing method for iron aluminide is necessary. In recent years, the wire-arc additive manufacturing (WAAM) process has obtained considerable progress in both manufacturing accuracy (through improved robotic path planning) [4,5], and application scope of materials, such as aluminum alloys, steel and titanium alloys [6–8]. Moreover, the WAAM process has already proved its capability of in-situ fabricating intermetallics of titanium aluminide [9,10] and iron aluminide [11,12] with controllable chemical compositions. Compared to traditional methods of producing iron aluminide, such as furnace casting/melting [13] and mechanical hot pressing [14], the WAAM process is capable of directly producing structures with full density which eliminates the need for expensive post-fabrication processing. Also, the cost of filler wires in the WAAM process is much lower than the high-purity metal powder which is necessary for powder metallurgical methods as used to prevent casting defects [15].

In order to further understand and develop the WAAM process, profound knowledge of the materials behavior during the multi-deposition process is required. During the process of additive manufacturing, the pre-deposited layer will be partially remelted by the next deposition process, and substantially reheated several times during the buildup. Specific to the buildup process of Fe_3Al based iron aluminide, the as-deposited material will experience phase transformation between Fe_3Al and FeAl every time the subsequent layer is deposited [16] as related to the phase diagram shown in Fig. 1, which will induce stress and influence the mechanical properties of the buildup structures.

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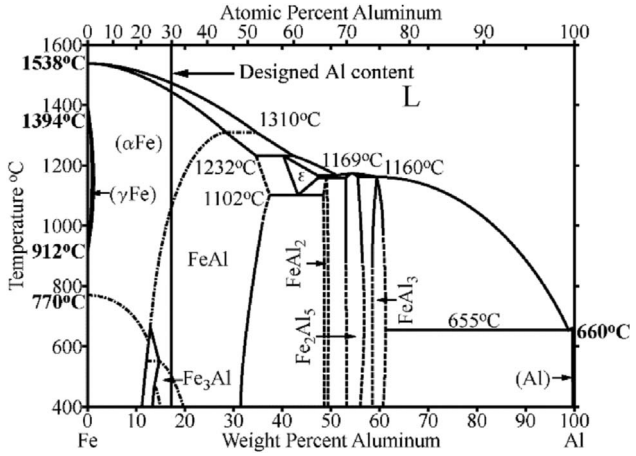

Fig. 1. Fe-Al binary diagram (the nominal composition of the investigated alloy is 30 at% Al).

Therefore, an in-situ observation and quantitative analysis of the phase transformation processes and grain structure variation are desired.

In the present research, the in-situ diffraction experiment was performed using the high-flux neutron diffractometer WOMBAT located on the TG1 thermal neutron guide at the Open Pool Australian Lightwater (OPAL) reactor. It is equipped with a 120° position sensitive detector, for which the major sensitive area is set up for high-speed recording and capable of measuring real-time phase transformations in seconds [17–22]. The neutron diffraction data were accumulated in 35 s time slices while the specimen was heated in a vacuum furnace to simulate the heat-up process during WAAM. Similar methods have been widely applied to investigate specific material properties in recent years [23–25]. Since both $D0_3$ structured Fe_3Al and B2 structured FeAl phases are ordered structures, their existence can be observed in superstructure diffraction peaks. Accordingly, the phase transformations can be quantitatively detected by the variation and disappearance of the corresponding peaks. Also, the grain structure variation occurring during the heat-up process can be analyzed using the obtained diffraction patterns.

2. Experimental setup

2.1. Sample preparation

The setup of the WAAM process is shown in Fig. 2. The process was powered using a gas tungsten arc welding (GTAW) arc which was generated by a commercial inverter power source and a matching (2.4 mm diameter) tungsten welding torch [11,12]. Two wire feeders with independent speed controls, one for 1080 grade aluminum wire (695 mm/min) and one for annealed high purity (99.99%) iron wire (1000 mm/min), were applied to feed the wires into a single molten pool and fabricate in-situ the designed 30 at% Al iron aluminide alloy. The substrate was chosen as 5 mm thick DH36 shipbuilding steel sheet, which possesses good weldability so as to ensure the stability of the deposition processes in the first few layers. The specific fabrication parameters chosen for the fabrication were 140 A deposition current, 673 K interpass temperature and 95 mm/min travel speed. The interpass temperature was maintained by clamping the substrate over a heating blanket placed in a thermal insulated box. The shielding gas chosen for the WAAM process is pure argon. In addition of the inert gas shielding of the GTAW torch itself, a trailing gas shielding cover was applied to maintain the gas shielding during the fast cooling process of the deposited material. The cuboid test specimen ($8 \times 10 \times 10$ mm^3) was extracted from the middle section of the buildup wall in order to avoid the effect of any dilution affected zone near the substrate. Considering the room temperature brittleness of Fe_3Al based iron aluminide, the cutting method chosen was wire electro-discharge machining.

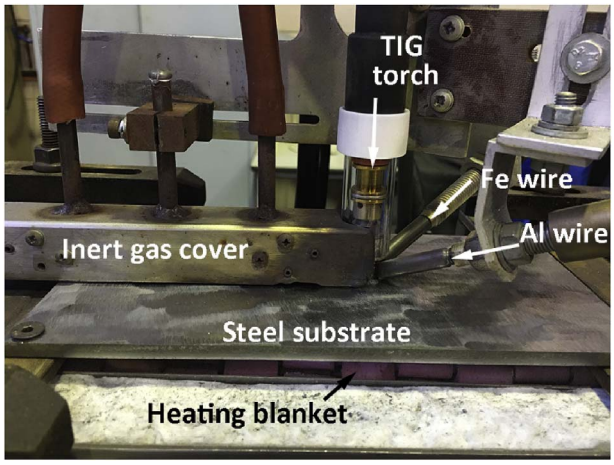

Fig. 2. Specific setup of the WAAM process.

2.2. In-situ neutron diffraction and data analysis

The WOMBAT diffractometer was calibrated by a LaB_6 standard to a wavelength of $\lambda = 2.419$ Å equivalent to a wavenumber $k = 2.597$ Å^{-1}. The 30 at% Al iron aluminide specimen was held in the center of the vacuum furnace (5×10^{-4} Pa) using molybdenum wire. Neutron data were collected to each data file every 35 s, which is a comparatively short acquisition time due to the high-intensity and data collecting features at WOMBAT. During the heat treatment, the temperature was first held at 305 K for 12 min in order to acquire the diffraction patterns at room-temperature with enough intensity and accuracy; then the temperature went up to 1623 K at a speed of 0.07 K/s. Subsequently, the temperature was held at 1623 K for 1200 s and cooled down at 0.07 K/s to 1373 K. Afterwards, in order to accelerate the cooling rate, the furnace was filled with pure helium gas and cooled to room-temperature. A C-type thermocouple was placed near the sample surface for reading and controlling the temperature. The heat treatment temperature profile is shown in Fig. 3.

In order to calculate quantitative phase fractions from the in-situ neutron data, Rietveld analysis was fitted to the diffractograms [26]. The obtained patterns were sliced into several featured sections according to the existence of certain peaks which would appear or disappear due to the phase transformations. Subsequently, five 1-D diffraction patterns from each featured section were imported to the Rietveld analysis software Bruker Topas V4.2 to determine the phase fractions. The structural models used included a cubic (Fm-3m, $D0_3$) Fe_3Al phase and a cubic (*Pm*-3m, B2) FeAl phase. A generalized spherical harmonics model was applied to account for the texture effect of

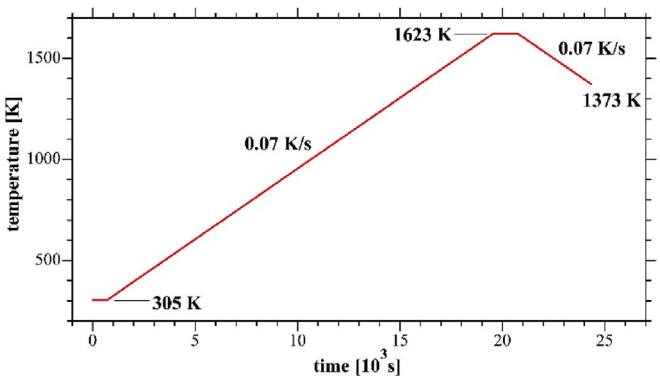

Fig. 3. Temperature profile of the heat treatment during the neutron data acquisition in frames of 35 s.