Journal of Alloys and Compounds 739 (2018) 712–722

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

Correlating microstructure and superelasticity of directed energy deposition additive manufactured Ni-rich NiTi alloys

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ARTICLE INFO

Article history:
Received 8 September 2017
Received in revised form
18 November 2017
Accepted 23 December 2017
Available online 29 December 2017

Keywords:
Metals and alloys
Laser processing
Microstructure
Mechanical properties
Shape memory
Scanning electron microscopy

ABSTRACT

Laser-based directed energy deposition (LDED) additive manufacturing of Ni-rich NiTi shape memory alloys was shown to produce inhomogeneous precipitate morphologies and characteristic grain structures consisting of columnar grains coexisting with equiaxed and subgrain structures. Post-processing solutionizing and aging heat treatments impacted microstructure and martensitic phase transformation (MT) responses underpinning superelastic shape memory responses. A solution treatment of 950 °C for 24 h was found to produce a uniform composition of the B2 austenite parent phase without affecting the coexistence of columnar and equiaxed substructures. Aging the solution treated material brought about a spatially uniform Ni_4Ti_3 precipitate morphology. Due to the uniform morphology, an underlying austenite-martensite interface motion accompanies the compressive stress-induced MT (SIMT). Reversible interface motion underpinned the compressive superelastic response for the solutionized and aged condition. On the other hand, strain concentrations existed at different spatial locations in the as built condition as well as when the as built material was aged. The stark contrasts in the SIMT exposed precipitate morphology as a controlling factor in tailoring the superelastic response of Ni-rich NiTi SMAs fabricated by LDED.

1. Introduction

Additive manufacturing (AM) of NiTi shape memory alloys (SMAs) has received significant attention recently [1,2]. The AM material microstructure interacts with the martensitic transformation (MT), which dictates the SMA behavior. Laser-based directed energy deposition (LDED) AM has the capability for tailoring the SMA response by controlling the microstructure at resolutions not possible through conventional NiTi processing methods [3]. The grain morphology produced by the LDED AM process can be tailored by altering the processing parameters [4–8] and using differential feedstock compositions to tailor the build composition [9,10]. The characteristic layer-by-layer deposition process in AM produces spatially varying thermal histories at each location within the build resulting in complex microstructures in as built materials [1,11,12]. Additional microstructural characterization at lengths scales known to affect the MT is needed. Post-process heat treatments are another route for tailoring transformation strain, phase transformation temperatures and critical transformation stress of as built NiTi AM materials.

During the AM process, local regions are remelted due to overlap between layers and passes, and the deposition of additional layers thermally treats previously solidified layers. As a result, the microconstituent phases, grains, and composition spatially vary as each location experiences a unique series of complex heating and cooling cycles. In previous works on AM NiTi, the presence of undesired secondary phases was found in locations closer to the substrate [13,14] and in the overlap regions between layers and passes [15]. Studies have shown that grains typically orient in the direction of the largest thermal gradient (z-direction) [2,16–20]. For AM builds, grain structures consist of characteristic columnar grains [2,16–19] and equiaxed [6,7] morphologies with varying sizes [4,7,15,17,18,21]. Compositional segregation within a solidified track may occur [17]. To elucidate spatial variations, the characterization of regions encompassing multiple layers and multiple passes is required, to, for example, correlate grain structure with the path used to deposit the material [2,15–18].

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https://doi.org/10.1016/j.jallcom.2017.12.270

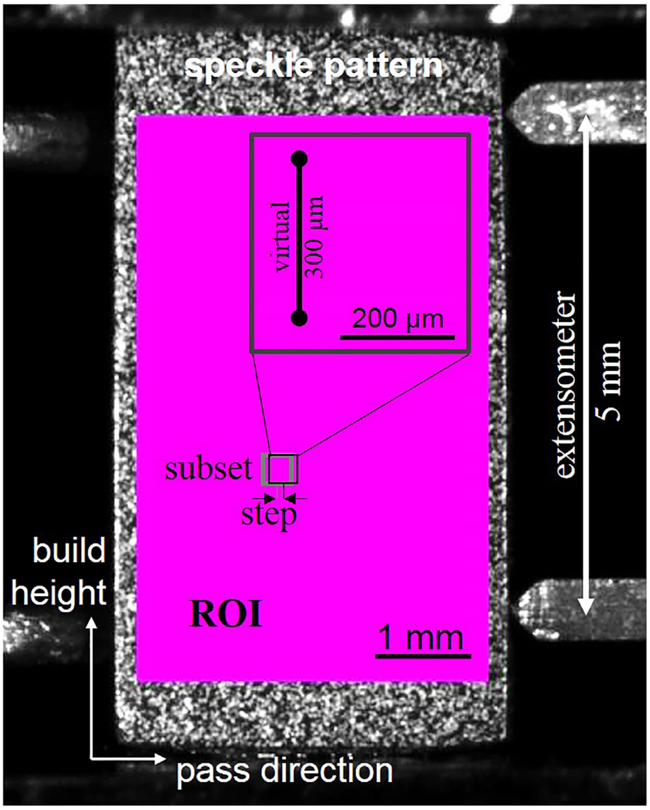

Fig. 1. Length scales for superelastic strain analysis. The extensometer (5 mm between legs) provides macroscale measurements. Compression specimen surface with the spray-painted speckle pattern shows parameters for DIC analysis including the region of interest (ROI), subset, and subset step size. The subset is magnified and inset onto the ROI with a finer scale bar (200 μm). The 300 μm DIC virtual gage is used to obtain contours in Fig. 9 (b), 10 (b), 11 (b), and 12 (b).

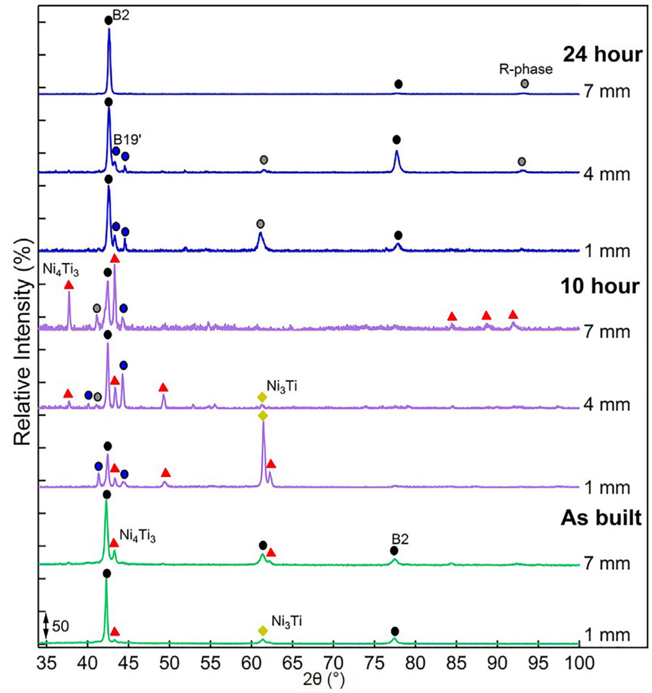

Fig. 3. XRD scans with increasing post deposition heat treatment duration at 950 °C. The specified locations are along the build height direction. Phases have been identified as B2 ●; B19' ●; R-phase ●; Ni_4Ti_3 ▲; Ni_3Ti ◆.

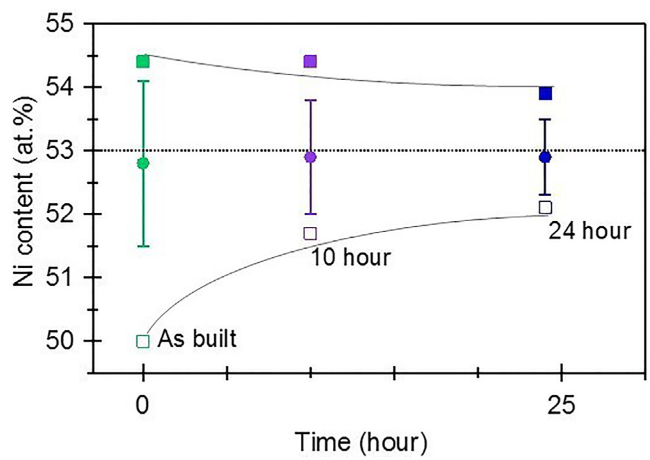

Fig. 4. Evolution of composition with increasing post deposition heat treatment duration at 950 °C. The dashed horizontal line is the input powder feedstock composition. Circles represent average compositions. Squares represent maxima and minima.

Post-deposition heat treatments typically consist of a high temperature solution treatment followed by a lower temperature precipitation aging treatment [2,14,15,19,22–24]. Solution treatment temperatures range from 800 °C to 1050 °C [25] are designed to homogenize the material and produce a uniform composition with no secondary phases [10,24]. For solution treatments at 950 °C, 5 h [15] and 1050 °C, 10 h [24] AM NiTi alloy microstructures have been found to still contain second phases. Studies are needed to confirm solution treatment temperatures and times that homogenize the material and produce a uniform composition with no secondary phases present. After solution treating AM NiTi alloys, grain sizes can increase, and grain substructures in overlap regions disappear [15]. Previous studies have correlated the solution

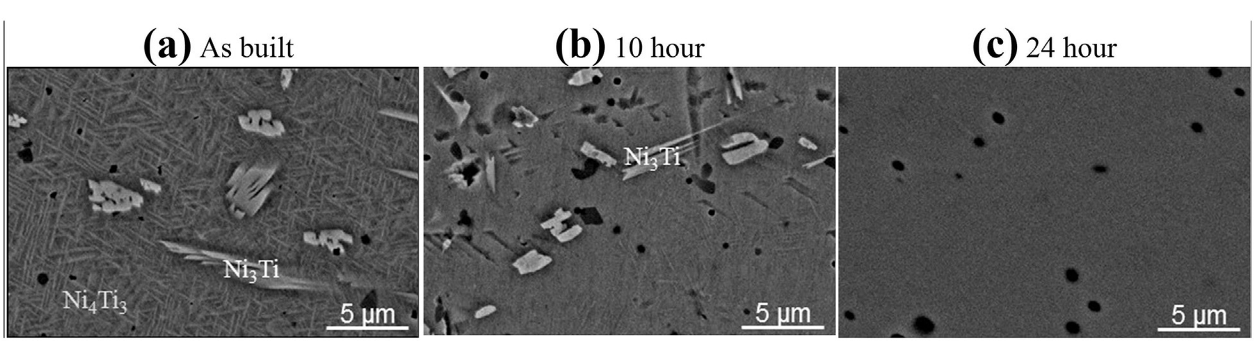

Fig. 2. Back scatter electron images showing the microconstituent morphologies for **(a)** as built alloys and alloys heat treated at 950 °C for **(b)** 10 and **(c)** 24 h durations. In **(a)** and **(b)**, lenticular microconstituents are Ni_4Ti_3 precipitates and Ni_3Ti secondary phases appear globular.