Isomorphic grain inoculation in Ti-6Al-4V during additive manufacturing

J.R. Kennedy [a],*, A.E. Davis [a], A. Caballero [b], A. Garner [a], J. Donoghue [a], S. Williams [b], J. Zollinger [c], E. Bouzy [d], E.J. Pickering [a], P.B. Prangnell [a]

[a] University of Manchester, Department of Materials, Manchester M13 9PL, United Kingdom
[b] Cranfield University, Welding Engineering and Laser Processing Centre, Bedfordshire MK43 0PL, United Kingdom
[c] Université de Lorraine, Institut Jean Lamour, Campus ARTEM, Allée André Guinier, F-54011 Nancy, France
[d] Université de Lorraine, CNRS, Arts et Métiers ParisTech, LEM3, F-57000 Metz, France

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ABSTRACT

The potential for using isomorphic inoculation (ISI) to grain refine titanium alloys in additive manufacturing was investigated by adding TiAlNb particles to Ti-64 during building test samples. A surviving particle was identified and its crystallographic relationship with the matrix studied by transmission Kikuchi diffraction. The particle and bulk matrix grain were shown to have the same crystallographic orientation, demonstrating that the ISI mechanism of solidification bypasses the nucleation step in favour of direct epitaxial growth.

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1. Introduction

In additive manufacturing (AM) the solidification structure is very important as there are typically few subsequent processing steps available to modify the microstructure. Solidification normally occurs by nuclei forming on heterogeneous sites in contact with the melt [1]. In the case of a moving melt pool, this can be at the fusion boundary or on particles suspended in the liquid, providing sufficient undercooling is achieved to overcome the energy barrier, which limits the number of nucleation sites [2]. The classical spherical cap model [1] does not adequately describe more potent sites, where nucleation is better treated by particle wetting [3] or layer-by-layer adsorption [4], nor situations where solidification is assisted by limited liquid ordering [5]. However, in all these cases solidification occurs by a two-step nucleation and growth process where there is a significant energy barrier to nucleation. In contrast, AM solidification can occur epitaxially from the fusion boundary with no energy barrier, frequently leading to coarse columnar grain structures in Ti alloys [6]. Recently, a novel grain refining method has been developed for cast TiAl based alloys, termed Isomorphic Inoculation (ISI), where the nucleation step is bypassed in favour of free growth directly from particles added to the melt, which are of the same phase as the solidifying material,

engineered to have a small lattice mismatch with the bulk and increased stability in the melt [7]. As there is a minimal energy barrier, in principle each particle can participate in solidification, providing it survives the melt [8]. However, since the particles and solidifying material are of the same phase, locating the surviving particle cores in the solidified material to confirm this mechanism is difficult, and has not been previously achieved. In this paper the feasibility of applying the ISI concept to grain refining titanium alloys in AM has been investigated, using Ti-64 with a Wire-Arc AM (WAAM) process. A surviving ISI particle was located and extracted by PFIB lift-out so that the particle/bulk interface could be investigated. It should be noted that on cooling the primary β phase that solidifies in Ti64 transforms to 95% α, which makes interpretation of the orientation relationship between the nucleation particle and matrix challenging. This limitation was overcome by using the Burgers orientation relationship (BOR) ($\{110\}_\beta//\{0001\}_\alpha$ and $\langle 111 \rangle_\beta//\langle 11\overline{2}0 \rangle_\alpha$) [9] to reconstruct the original β parent orientation and exploiting high resolution orientation mapping with transmission Kikuchi diffraction (TKD).

2. Materials and methods

Critical to the ISI method is to ensure the lattice mismatch between the particles and the solidifying matrix are as small as possible, minimizing the interfacial energy to induce epitaxial growth. To this end, the lattice parameters of the Ti-10Al-25Nb

* Corresponding author.
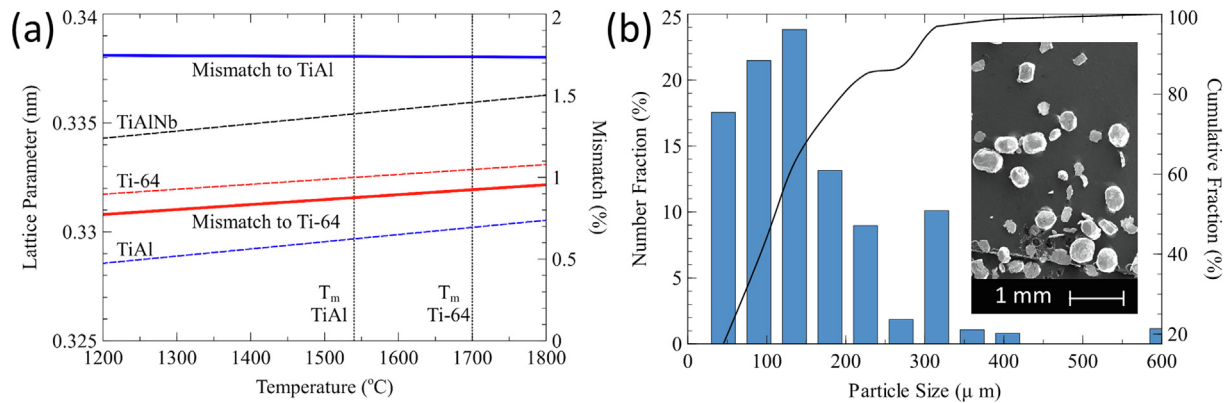E-mail address: jacob.kennedy@manchester.ac.uk (J.R. Kennedy).

Fig. 1. (a) Lattice parameters and matrix alloy mismatch for ISI Tl10Al25Nb particles relative to Ti46Al and Ti-64, near the melting points of both alloys, and (b) the size distribution of TiAlNb particles added to the WAAM process with inset SEM image.

Table 1
The WAAM plasma-arc deposition parameters.

Travel Speed	5 (mm s^{-1})	Layer Height	~1.1 (mm)
Current	160 (A)	Plasma Gas Flow	0.013 (L s^{-1})
Wire Diameter	1.2 (mm)	Wire Feed Speed	2 (m min^{-1})

(TiAlNb) ISI particles, previously used successfully with Ti-46Al castings [7,8], were compared with Ti-64 (calculated using high temperature β-phase lattice parameters [10] and the coefficient of thermal expansion [6]). At the melting point of the bulk alloys, the β-BCC (body-centred cubic) phase formed by TiAlNb was predicted to have less than a 1% lattice mismatch with Ti-64 (Fig. 1 (a)), less than that of the previously used TiAl alloy, indicating that it should be suitable as an ISI. However, as Ti-64 has a higher melting point (~1700 °C) than Ti-46Al (~1540 °C), the TiAlNb particles (~1800 °C) will be less stable. The ISI particles were prepared as described in [7] with the size distribution shown in Fig. 1(b).

To demonstrate the feasibility of ISI with Ti-64 during WAAM a single track (~9 mm) wide wall was built, 20 layers (~22 mm) high and 140 mm long (detailed in Table 1) in an argon atmosphere. The TiAlNb ISI particles were incorporated into the melt pool by adhering them to the surface of each layer, premixed with polyurethane (similar to [11]). Multiple cross sections were metallographically prepared and analyzed using a Zeiss Sigma SEM. After locating a surviving ISI particle, a Thermo Scientific Helios G4 P-FIB UXe, with an Oxford Instruments (OI) Symmetry EBSD detector, was used to lift-out a slice through the particle/matrix interface and thin the sample to electron transparency. The crystallographic orientations across the sample where then mapped by TKD at and analysed using the OI AZTEC software package.

3. Results and discussion

The surviving ISI particle was identified from its high Nb content by BSE imaging polished sample sections (Fig. 2a). With conventional EBSD, the matrix grain and particle core were mapped, but the interfacial diffusion boundary layer (~10 μm thick) did not index due to the fine-scale of the α transformation structure reducing pattern quality (Fig. 2b). As expected from its high Nb content (Fig. 2d), the ISI particle was fully β stabilised and contained no transformation structure (Fig. 2e). BOR β-reconstruction however allowed comparison of the orientations of the directly indexed particle core and the far-field parent β grain. These orientations are depicted in the Pole figures in Fig. 2g, which show that the particle and far-field matrix have identical crystal orientations. This can also be seen from the coloration in the IPF β phase orientation map in Fig. 2f. In addition, the α phase pole figures for the matrix in Fig. 2g, show clear alignment between the $\{110\}_\beta//\{0001\}_\alpha$ planes and $\langle 111 \rangle_\beta//\langle 11\overline{2}0 \rangle_\alpha$ directions expected by the BOR between the particle and surrounding transformed α variants that were indexed in the EBSD map.

The identical far field orientation between the ISI particle and matrix grain provides strong evidence that solidification occurred by epitaxial growth from the particle, however the unindexed boundary layer around the particle makes it difficult to be unequivocal. In order to analyse the interfacial region a section was removed by P-FIB lift-out, thinned and further characterized using TKD. Mapping the interfacial region at higher resolution (Fig. 3) shows a gradual transition from the fully β Nb rich particle to ultra-fine α, to the larger α laths in the surrounding Ti64 matrix, with the α volume fraction increasing as the Nb content diminishes. The transformed α laths also penetrate into the partially dissolved particle, rather than their being a sharp interface, as would be expected from classical heterogeneous nucleation from an inoculant [1,3,4]. Furthermore, TKD mapping of the interfacial region (Fig. 3b) shows that the directly indexed residual β extending into the boundary layer has the same orientation as the matrix β orientation reconstructed from the transformed α phase. It is thus evident from this progressive transition and the identical β orientations between the particle and matrix that solidification occurred, not by a heterogeneous nucleation event, but by direct epitaxial growth from the particle.

4. Conclusions

An experiment was conducted to test the concept of using a high melting point β-stabilized TiAlNb alloy powder to act as an isomorphic inoculant in Ti64 alloy during AM. Using the TKD technique, it has been shown that a surviving ISI particle located in the deposit had an epitaxial relationship to the matrix primary-β grain that formed on solidification. This confirms that the mechanism of solidification was by direct epitaxial growth with a minimal energy barrier and provides strong evidence that the isomorphic inoculation concept is viable in AM processes with Ti alloys.

The application of ISI to grain refining in AM has several advantages over conventional inoculant compounds [11]. They are more efficient than second phase compounds and can operate at near zero undercooling. Furthermore, they will be less mechanically damaging than conventional grain refiners. They can also be selected and used at an addition level where they do not prevent recycling of scrap streams.
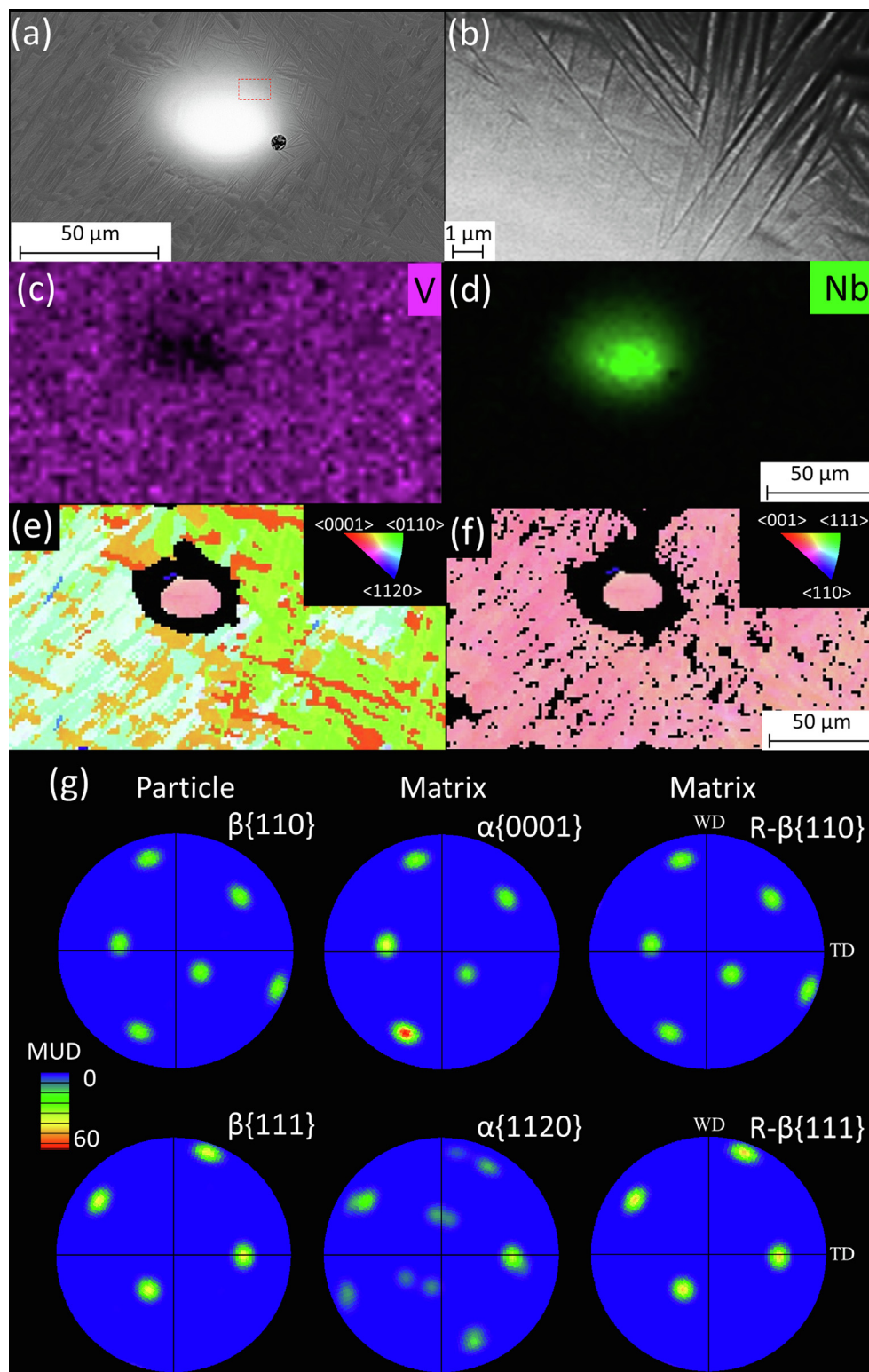
Fig. 2. BSE images of (a) particle region and (b) interface region highlighted using red box in (a), (c) EDS V and (d) Nb maps, (e) IPF ND α, and (f) reconstructed β matrix orientation maps, (g) particle pole figures (β), matrix (α), and parent reconstruction (R-β).
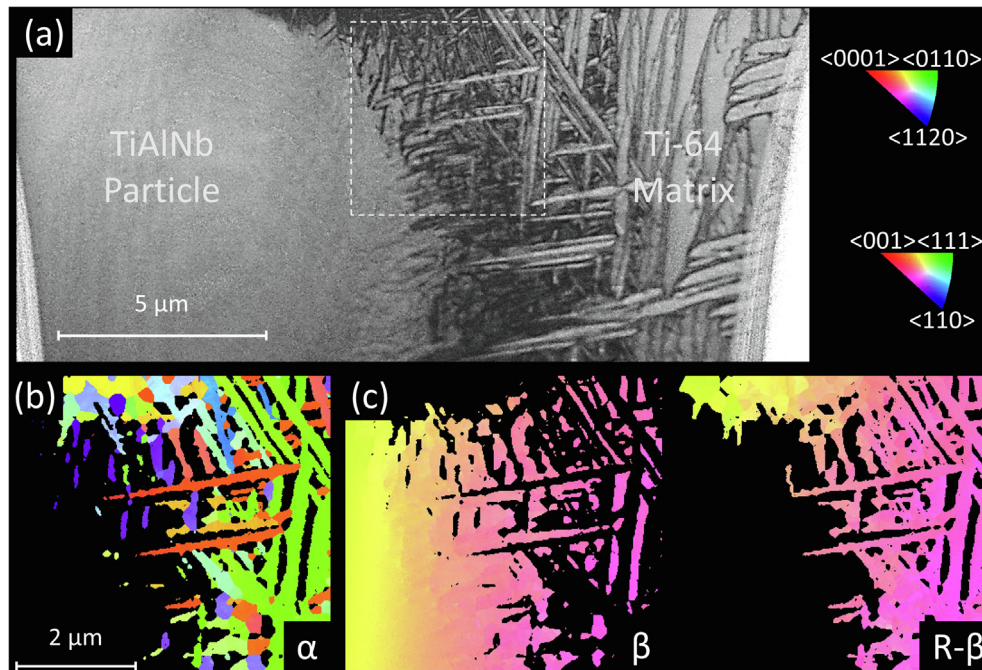
Fig. 3. TKD Investigation of particle/matrix interface; (a) band contrast image across the interfacial region, (b) TKD IPF ND orientation maps of the indexed α, and (c) a composite image showing the directly indexed β associated with the particle (left) and the reconstructed parent β orientation in the matrix (right).

A technical challenge remaining is how best to add the inoculating particles and control their survival during the brief high temperature exposure in the melt pool, to ensure a consistent level of grain refinement within the deposited metal.

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.

Acknowledgements

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