Direct Laser Additive Manufacturing of TiAl Intermetallic Compound by Powder Directed Energy Deposition (DED)

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Abstract

Directed Energy Deposition of the commercial intermetallic Ti-48Al-2Cr-2Nb alloy was investigated. The CLAD® process is dependent on multiple parameters, which were successfully optimised through several experiments, including series of beads, small blocks, and massive blocks, under argon atmosphere. The use of adapted temperature management leads to massive blocks manufacturing that bear no apparent macroscopic defects, such as cracks, which are generally observed in this brittle material due to strong temperature cycling during the manufacturing. The microstructure and geometrical parameters were characterised by scanning electron microscopy (SEM). This process generates an ultra-fine and anisotropic microstructure, which is restored to a homogeneous duplex microstructure by a subsequent heat-treatment. Mechanical characterisation is in progress and will be used to validate the soundness of the materials produced in these conditions.

Introduction

In the context of cost reduction, the increase of maximum working temperatures of aircraft engines is a way to improve their energy efficiency. This purpose drives users to develop high temperature alloys, such as gamma titanium aluminides. These materials are good candidates to replace nickel-based superalloys in the low-pressure turbine of the aircraft engines due to their low density, high Young's modulus and mechanical properties up to 800°C, as well as good hot corrosion resistance [1- 6].

However, their manufacturing through conventional processing route implies strong limitations on the achievable geometries, which could be overcome by a direct additive manufacturing process such as Powder Directed Energy Deposition [7]. The use of such a process already allows large scale complex functional parts made of titanium alloys [8- 12], nickel-based superalloys [13, 14] and several steels [15-19] to be manufactured.

Unfortunately, gamma titanium aluminides are known to present a high cracking susceptibility and low thermal shock resistance [7], which constitute key issues for the processing of this intermetallic compound.

Therefore, the control of thermal gradients during and after metal deposition is a key parameter to achieve defect-free materials and parts. The objective of this work is thus to study the deposition of a particular TiAl alloy onto a preheated substrate, using a high-temperature plate with integrated control of the heating and cooling rates.

Materials and equipment

Materials

The material used in this study is a first generation GE4822 alloy, produced by gas atomisation and sieved by LPW-UK. After sieving, the powder size distribution is within the 40–110µm range. The material's composition is detailed in Table 1.

	Ti	Al	Cr	Nb	C	N	O	Fe
% _{mass}	59.0	33.74	2.6	4.58	0.012	0.002	0.05	0.04
% _{atomic}	47.6	48.34	1.9	1.91	0.039	0.006	0.1	0.03

Table 1: Mass and atomic composition of the Ti-48Al-2Cr-2Nb intermetallic compound

The substrates are composed of Ti64 sheets with a thickness in the range 3–8mm, which are sandblasted prior to deposition trials.

Heating systems

Two heating systems were used. One is a commercial radiant plate with no temperature and rate controls, whereas the other one is an experimental resistive plate with temperature and rate controls, set-up by ONERA.

The temperatures on the substrates and deposited material are measured using K-type thermocouples.

Laser and deposition system

The parts were manufactured with the DED-CLAD® blown powder additive manufacturing process. The additive manufacturing machine is equipped with a coaxial nozzle (developed by IREPA LASER, US patent n°5418350).

A 980nm wavelength 2kW laser diode was used with a 600µm fibre diameter. The focal point position was set at a 12.5 mm distance under the lowest part of the nozzle. The laser beam had a 2.2mm diameter at the focus point and presented a homogenous "top hat" energy distribution.

Inert gas chamber

Due to the high affinity of γ-TiAl compound with oxygen, manufacturing processes were carried out under argon gas. The measured oxygen and water content were kept under 10ppm and 50ppm, respectively, thanks to the inert gas chamber purification system.

Micrographic preparations

The samples were cut by wire EDM in order to analyse the microstructure of the deposited material and to produce some mechanical testing samples.

Before microstructural observations, the sintered materials were polished by standard method, using different SiC grinding papers up to grade 2400, then with 3 μ m and 1/4 μ m diamond paste and finally with a 3h-long vibratory finishing in colloidal silica gel.

The microstructures were observed by scanning electron microscopy (ZEISS DSM 962) and chemical analysis was done by energy dispersive X-ray spectrometry (SEM-EDS).

Thermal treatment

Before mechanical testing, the test samples are heat-treated under argon to homogenise the microstructure. It is well known that homogeneous microstructures are preferred to guarantee a satisfactory level in mechanical properties. The successive steps are:

- 4 hours annealing at 1250°C, just above their $\alpha/\alpha+\gamma$ transformation
- air quenching
- 4 hours tempering at 900°C, in the $\gamma+\alpha_2$ region
- slow natural cooling in the furnace

Experimental procedure

Two series of experiments were performed to identify the processing parameters leading to fully dense and sound samples. A first set of experiments was carried out on single beads, in order to determine the individual processing parameters in which satisfactory geometries can be obtained. A second set of experiments was then set-up in order to validate these building parameters, before building blocks for mechanical testing.

Single beads deposition

A first screening is carried out by depositing simple, 90mm-long, beads with several parameter sets, including scan speed, powder mass flow rate (MFR) and laser power. The parameter ranges are detailed in Table 2 below.

Parameter	Min value	Max value
Deposition speed (m/min)	0.5	2
Powder mass flow (g/min)	8.8	14.5
Laser power (kW)	1.2	1.8

Table 2: Parameter ranges for the first deposition run

The substrates are 8mm-thick and the heating plate is the commercial radiant plate. The achieved temperatures on the surface of the substrates are measured in the range 400–430°C for the first one and in the 360–400°C range for the second one. Besides, no control of the heating and cooling rates was possible with this equipment.

Figure 1: Optical photograph of the simple beads deposited in first screening

Bead n°	Preheating temperature (°C)	Laser Power (W)	Powder mass flow rate (g/min)	Deposition speed (mm/min)
1-4	400-430	1500	8.8	2000
5-8	400-430	1500	11.7	2000
9-10	400-430	1500	14.5	2000
14	360-400	1500	14.5	2000
15	360-400	1500	8.8	1500
16	360-400	1500	8.8	1000
17	360-400	1500	8.8	500
18	360-400	1500	11.7	1500
19	360-400	1500	11.7	1000
20	360-400	1500	11.7	500
21	360-400	1500	14.5	1500

22	360-400	1500	14.5	1000
23	360-400	1500	14.5	500
24	360-400	1800	14.5	1000
25	360-400	1200	14.5	1000

Table 3: Deposition parameters of the first TiAl deposition series

Exploratory blocks building

A second series of tests is run in order to build small 50mm-long blocks (4 beads wide, 10 layers high). The sets of parameters used for these runs are chosen from the first single beads that showed the lowest number of macrographic cracks (none or only one). These sets use fixed laser power and powder MFR, and three levels of scan speed (see Table 4).

Parameter	Values		
Parameter set reference	P1	P2	P3
Deposition speed (m/min)	0.5	1	1.5
Powder mass flow (g/min)	8.8		
Laser power (kW)	1.5		

Table 4: Parameter values for the second deposition run

The substrate is 3.2mm-thick and the heating plate is ONERA's resistive plate. The heating elements dwell temperature is 1100°C and the temperature achieved on the surface of the substrates is measured in the range 370–420°C. The heating and cooling rates of the resistive elements are respectively 20°C/min and 2°C/min. The substrate's cooling rate has previously been evaluated around 0.8°C/min in these cooling conditions, with the use of a thermal camera.

Blocks for mechanical testing

In order to machine test samples for mechanical testing, a third series – based on the results of the previous run – was manufactured. For each selected parameter set, a block and a wall were built. Blocks dimensions are over 50x40x25 mm³ (LxWxH) in order to machine X and Y oriented samples; walls dimensions are over 50x10x40 mm³ (LxWxH) in order to machine Z oriented samples.

The two parameters sets with the smallest scan speeds were selected, in order to reduce the number of trials. The number of layers and of beads per layer was set to obtain the desired dimensions (Tables 5 and 6).

Blocks	# beads per layer	Aimed width (mm)	# layer per block	Aimed height (mm)
P1 (0.5m/min)	12	37.4	25	31.5
P2 (1m/min)	16	39.1	35	29.1

Table 5: Number of beads and aimed dimensions for the XY-blocks

Walls	# beads per layer	Aimed width (mm)	# layer per wall	Aimed height (mm)
P1 (0.5m/min)	4	13.3	35	44.1
P2 (1m/min)	4	10.5	50	41.5

Table 6: Number of beads and aimed dimensions for the Z-walls

The substrates are 6mm-thick and the heating plate is the commercial plate, due to unavailability of ONERA's one. Therefore no control of the heating and cooling rates was achievable. The achieved temperature on the surface of the substrates is measured in the range 215-230°C. This strong decrease in temperature is not explained, considering that the equipment is identical to the one used in the first series, and that a thinner substrate is used, which should have increased the measured surface temperature.

Blocks with heat treatment for mechanical testing

Due to very low preheating temperature and lack of control of the cooling rates in the previous run, a fourth series of blocks manufacturing were undertaken. These experiments were performed with the ONERA heating plate and an insulating lid was designed in order to help preheating to a higher temperature as well as postheating in a better controlled thermal environment.

This series consists in XY-blocks only; the three deposition parameters sets are the ones from the second series (P1 to P3, see Table 4). For each set, three cooling conditions are operated, for a total number of 9 experiments. The three cooling conditions are described hereafter:

- C1 Air quenching on a refractory brick at ambient temperature, just after manufacturing
- C2 Reheating for 30 minutes under the insulating lid
+ air cooling in a thermally insulating box
- C3 Reheating for 30 minutes under the insulating lid
+ 2°C/min controlled cooling on the heating plate, under insulating lid

Preheating temperatures were measured just before the removal of the lid. They are reported in Table 7, hereafter:

	T _{start} (°C)
P1C1	930
P1C2	915
P1C3	905
P2C1	960
P2C2	960
P2C3	925
P3C1	896
P3C2	900

P3C3 890

Table 7: Preheating temperature for all experiments of the fourth series

The P2C1 build temperature evolution has also been measured throughout the experiment, thanks to a K-type thermocouple welded on the substrate at a position detailed in Figure 2.

Figure 2: Photograph of the P2C1 build with positioning of the thermocouple with regards to the substrate

Moreover, in order to release the stresses before machining of the test samples, a supplementary heat treatment with the following characteristics was carried out on each build after its complete cool down:

- Static air atmosphere
- 600°C for 2 hours
- 10°C/min heating rate
- Slow in-furnace cooling down

Results and discussion

Single beads analysis

The as-built single beads microstructure shows a complete crystallisation and very few defects such as unmelted particles and porosities. However most of the beads show a high number of transverse cracks, which formed during fast cooling, just after the deposition (Figure 3). This high crack density is attributed to the combination of the high brittleness of the material, the strong temperature decrease after deposition and the non-deformation of the 8mm-thick substrate.

Figure 3: Number of cracks per bead deposited in the first series

However, some beads show a seemingly low number of transverse cracks. These are the beads n° 1-3 and 15, 16 and 17. It appears that these specific beads were deposited under a 1500W laser power and a powder MFR of 8.8g/min. The scan speeds were 2, 1.5, 1 and 0.5m/min respectively. This low number of cracks could be correlated to the high power/MFR ratio, compared to all other samples.

Figure 4: Secondary electrons micrographs of a transverse crack in the bead 18 of the first series

Only the second substrate (beads 14 to 25) was further investigated by SEM due to the high number of similar beads on the first one.

Figure 5: Backscattered electrons micrographs of 1.5kW – 8.8g/min beads build at 0.5, 1 and 1.5m/min (17, 16 and 15 respectively)

Figure 6: Backscattered electrons micrographs of 1.5kW – 11.7g/min beads build at 0.5, 1 and 1.5m/min (20, 19 and 18 respectively)

Figure 7: Backscattered electrons micrographs of 1.5kW – 14.5g/min beads build at 0.5, 1, 1.5 and 2m/min (23, 22, 21 and 14 respectively)

Figure 8: Backscattered electrons micrographs of 1m/min – 14.5g/min beads build with 1.2 and 1.8kW (24 and 15 respectively)

Bead Ref.	Height (mm)	Width (mm)	Height/width ratio	Dilution (mm)	Height/dilution ratio
14	0.63	2.8	0.23	0.21	3
15	0.64	2.8	0.23	0.4	1.6
16	0.83	3.4	0.24	0.35	2.37
17	1.4	4.3	0.33	0.38	3.68
18	0.71	3	0.24	0.27	2.63

19	1.03	3.3	0.31	0.28	3.68
20	1.67	4	0.42	0.26	6.42
21	0.84	3	0.28	0.24	3.5
22	1.2	3.2	0.38	0.2	6
23	1.9	4	0.48	0.1	19
24	1.23	3.8	0.33	0.3	4.1
25	1.1	2.9	0.39	0.1	11

Table 8: Geometrical measurement of the beads 14 to 25, measured on previous electron micrographs

Most of these beads show few unmelted particles and porosities, as well as a high dilution, with a depth comprised between 200 and 400 μ m. Only the beads 23 and 25 show a bad substrate interface, with a wetting angle over 90°, and a very low penetration depth in the substrate (100 μ m). Moreover, bead 23 shows numerous defects, probably due to a lack of particle melting, which could be caused by the combination of a high powder MFR (14.5g/min) and a low scan speed (0.5m/min).

Considering the non-optimised thermal building condition, some of these first tests show interesting results, regarding both the number of defects and the bead morphology. Therefore, the deposition parameters of beads 15, 16 and 17 were retained for the next step, dealing with beads stacking in order to build small blocks.

This next step was carried out with the heating plate developed by ONERA, which contributed in controlling the heating and cooling rate of the substrate and builds.

Blocks from series 2 and 3

Optical photographs of the builds from the second and third series are presented in Figures 9 and 10, respectively.

It is quite clear that the thin substrate (3.2mm) of the second run is strongly bent and has accommodated the build's stresses. It is believed that such strain of the substrate could explain the absence of visible cracks on the blocks and the good cohesion of the TiAl layers with the substrate.

Figure 9: Optical photographs of the small exploratory blocks (second series)

Quite expectedly, the thicker substrate (6mm) of the third run is less bent and the strain mainly occurs under the XY-blocks, as its contact area with the substrate is more important as the one of the Z-walls.

Cracks appeared in the P1 XY-block during the cooling down (Figure 10, left). This defect is attributed to the very low preheating temperature and the uncontrolled cooling rate of the samples. The cracking of the P2 XY-block during the machining of the sample (Figure 10, right) is also attributed to these issues. Due to the lesser contact area with the substrate and the shorter processing time for one layer, cracking was avoided during the Z-walls processing.

From a macroscopic point of view, all the blocks from these two series appear to be homogeneous and compact, and very few unmelted particles can be observed on the surface.

Figure 10: Optical photographs of the XY-blocks and Z-walls from third series (0.5mm/min, left – 1mm/min, right)

The SEM images of the second series (Figures 11 to 13) clearly shows some small lacks of matter, which only appear close to the surface. These are formed in the last layer, which has not been re-melted by a laser scan.

The as-deposited microstructure of all builds from second and third series is highly anisotropic, with no evidence of interface porosity. Dendritic solidification structures underline the bottom of each bead, whereas a finer microstructure is visible on their top part.

The black dots that are noticed on all samples are not attributed to porosity, but to a carbon contamination during the preparation of the samples, as they are also spotted on the Ti64 substrate that is, undoubtedly, fully dense.

Figure 11: Backscattered electrons micrographs of the first (P3) small block, build with a 1.5m/min deposition speed

Figure 12: Backscattered electrons micrographs of the second (P2) small block, build with a 1m/min deposition speed

Figure 13: Backscattered electrons micrographs of the third (P1) small block, build with a 0.5m/min deposition speed

Figure 14: Backscattered electrons micrographs of as-deposited P1 (left) and P2 (right) XY-blocks at low magnification (x50)

However, SE observations of the XY-blocs at higher magnification (Figures 15 and 16, right) exhibit some μm -size defects that look like shrinkage defects. These are only visible in SE mode and do not appear in BSE mode (Figures 15 and 16, left). Such defects might have already been present in the exploratory blocks, but were

not detected as these samples were only observed in BSE mode.

Figure 15: Defect observation P1 XY-block – backscattered electrons (left), secondary electrons (right)

Figure 16: Defect observation P2 XY-block – backscattered electrons (left), secondary electrons (right)

All low-magnification SEM observations (Figures 11 to 14) also show the presence of a few ‘unmelted’ particles in both series, with all three parameter sets. They are highlighted by their lighter colour in BSE mode, which indicates that their composition is enriched in heavy element, such as titanium.

Figure 17: Backscattered electrons micrographs of a lighter-colour particle observed in the P2 XY-block (third series)

The EDX spectroscopy of one of the lighter particles (Figure 17) confirms the Ti-enriched composition, with an estimated 60-80% atomic content in Ti (Table 9).

Spot n°	Al (% _{atom})	Ti (% _{atom})	Cr (% _{atom})	Nb (% _{atom})
1	39	58	1,4	1,6
2	33	65	1	0,5
3	20	78	1,6	0,4
4	41	56	1,5	1,6
5	32	66	0,8	0,6
6	47	49	2,5	1,1
7	44	52	2,1	1,7

Table 9: Estimated atomic composition of a lighter particle in the P2 XY-block (third series), measured by semi-quantitative EDS

However, a careful examination of Figure 17 reveals a solidification microstructure, similar to the one found in the rest of the build. This indicates that they are not unfused particles, but that their composition differs from the rest of the build and that the melt pool lifetime is probably not long enough to allow diffusion and achieve chemical homogeneity.

An additional BSE observation of the powder before deposition revealed the presence of particles that are also enriched in heavy element in the initial powder (Figure 18).

The EDX spectroscopy of these lighter particles confirms the Ti-enriched composition, with an estimated 90% atomic content in Ti (Table 10). This observation can explain the presence of unmelted particles in the build, as the melt pool lifetime is not long enough to allow for the complete homogenisation of these Ti-enriched particles by diffusion with the rest of the TiAl build.

Figure 18: Backscattered electrons micrographs of the TiAl powder before deposition

	Al (% _{atom})	Ti (% _{atom})	Cr (% _{atom})	Nb (% _{atom})
Light particles	9,5	90,1	0,4	0
	9,4	90,2	0,4	0
Dark particles	46,8	49,5	2,1	1,5
	47,5	49	2,2	1,3

Table 10: Estimated atomic composition of light and dark particles, measured by semi-quantitative EDS

In order to homogenise the as-deposited anisotropic microstructure, the previously mentioned heat treatment was applied to the test samples cut in the third series before mechanical testing. The same procedure was also applied to the small blocks of the second series which were deposited with the same parameter sets (P1 and P2), in order to compare the effect of heat treatment on their microstructure (Figures 19 to 22).

Figure 19: Backscattered electrons micrographs of as-deposited P1 small block (second series) at various magnifications

Figure 20: Backscattered electrons micrographs of heat-treated P1 small block (second series) at various magnifications

Figure 21: Backscattered electrons micrographs of as-deposited P2 block (1.5kW – 1m/min – 8.8g/min) at various magnifications

Figure 22: Backscattered electrons micrographs of heat-treated P2 block (1.5kW – 1m/min – 8.8g/min) at various magnifications

The as-deposited dendritic microstructure is transformed into a nearly uniform duplex microstructure containing $\gamma + \alpha_2$ lamellar colonies and γ grains, and erases the few Ti-rich particles by activating their diffusion in the stoichiometric TiAl matrix.

However, no information on the effect of this heat treatment on the μm -size shrinkage defect is available, as no SE observation was carried out on the heat-treated samples.

Mechanical properties are currently being tested for the two deposition parameter sets P1 and P2 through tensile testing. For each parameter set, the three directions are being investigated, with two test samples per direction.

Blocks with heat treatment for mechanical testing

The manufactured blocks of the fourth series show dimensions close to the desired values and appear crack-free on the macroscopic scale (Figure 23). The substrate shows a much smaller bending than on the previous runs, probably due to its larger thickness.

This absence of macroscopic defects in the build is attributed to the use of ONERA's heating plate, as well as of the insulating lid, and to the better thermal control that they provide.

At this stage, no difference is observed between the specimens cooled down in the various cooling conditions.

However, some tearing is observed at the junction of the build's edge with the substrate (Figure 24). This tear-off effect was not observed on the previous builds and is attributed to the lesser deformation of the substrate, which leads to stress relaxation by tearing instead of straining.

Figure 23: P2C3 block (1,5kW – 1m/min – 8,8g/min – controlled cooling down)

Figure 24: Tear-off effect at the junction with the substrate of block P2C3

The temperature measured during the P2C1 experiment is reported in Figures 25 and 26. These graphs show a fast decrease in temperature from 980 to 830°C in the minute required between the lifting of the lid and the beginning of the laser deposition.

A slower temperature decrease is observed in the first five layers. The temperature then stabilises at about 760°C for the next 15 layers and subsequently decreases again down to 740°C during the last 20 minutes.

It is interesting to note a strong decrease of up to 60°C at the beginning and end of each layer, *i.e.* when the deposition head moves back to the starting point of the next layer. This is attributed to the effect of forced convection induced by the powder carrier gas that still continues to flow between the layers.

Figure 25: Measured substrate surface temperature during the total P2C1 experiment

Figure 26: Measured substrate surface temperature during the P2C1 block building

These graphs show that this block was manufactured in the 825-725°C temperature range, which is in the range of the brittle/ductile transition temperature (750°C) [7] and is therefore beneficial for the build quality. Nonetheless, at the end of its manufacturing, the block goes through a steep temperature decrease that might increase its internal stresses, with a potential detrimental effect on the final mechanical properties.

Summary and outlook

In this study, several 60cm³-blocks of sound and dense γ -TiAl intermetallic compound were successfully manufactured by the DED-CLAD® direct additive process, despite the low preheating temperatures.

The apparition of macroscopic defects, such as cracks, was avoided thanks to an adapted preheating system, which allows working in the temperature range of ductile/brittle transition.

Some Ti-rich zones nonetheless appear in all the builds. They are attributed to the presence of Ti-rich particles in the initial powder, and hopefully do not question the deposition parameters.

The temperature characterisation of one build has confirmed the fast temperature decrease when the confined preheating area is opened to start the deposition. It has also evidenced the effect of the forced convection of carrier gas on the surface of the substrate.

All three selected deposition parameter sets led to a sound microstructure. However, this as-deposited microstructure is anisotropic due to dendritic solidification structures at the bottom of each bead. A fine and isotropic microstructure was obtained thanks to a subsequent thermal treatment.

Three different cooling conditions were tested, all of which allowed avoiding the occurrence of macroscopic cracks.

The effect of the low-temperature thermal post-treatment on the machinability of the blocks still has to be evaluated.

Some tensile test samples were machined in the third run blocks and walls and the mechanical properties of these builds are currently under characterisation.

This work has allowed us to make a first step towards the direct additive manufacturing of TiAl larger structural components and opens the way to some future development on the additive manufacturing of more advanced and more challenging intermetallic compositions.

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