

Direct Metal Deposition by Laser in TiNi-Al System for Graded Structure Fabrication

I Shishkovsky^{1,2}, N Kakovkina¹, F Missemmer²

¹ P.N. Lebedev Physical Institute (LPI) of the Russian Academy of Sciences, Samara branch, 443011 Samara, Russia

² Ecole Nationale d'Ingénieurs de Saint-Etienne, 58 rue Jean Parot, 42023 Saint-Etienne, France

E-mail: shiv@fian.smr.ru

Abstract. Intermetallic phase formation was studied in powdered TiNi-Al system under layerwise laser cladding with the aim of forming a gradient of properties due to a change in the concentration relation of Al in the NiTi powder mixture from one layer to another. The relationship between the laser cladding parameters and the intermetallic phase structures in consecutively cladded layers were determined. The structure of intermetallic compounds formed by laser synthesis was studied by optical microscopy, measurement of microhardness, SEM with EDX analysis. Microhardness doubling from 500 HV to 1000 HV was achieved due to nitinol matrix enrichment by Al, which is promising for aerospace applications.

1. Introduction

The application range of nickel superalloys is diverse and covers gas turbines for electro- or gas-pumping stations, rocket engines, automatic spacecraft and nuclear reactors. The basic units of turbines where nickel-based super alloys could be used are combustion chambers, guide blades in nozzles, rotor blades or turbine disks. TiAl-based intermetallics also have a number of advantages over the conventional titanium alloys, such as a higher modulus of elasticity, lower density, better mechanical properties at high temperatures. The study of peculiarities of phase structure transformations of TiNi-Al alloys (including those doped with aluminum), which can affect mechanical properties of products made by additive technologies using these powders, is an urgent problem. Due to such complex intermetallic phases, they could replace refractory nickel superalloys in future.

There are five mechanisms for strengthening superalloys: solid solution, dispersion, grain-boundary, deformation and textural strengthening [1]. The first three ones depend on the alloy nature. In nickel-based alloys the strengthening occurs essentially by the dispersive mechanism due to the release of γ -Ni₃(Al,Ti) phase. In our paper [2], laser beam (LB)-aided control of self-propagating high-temperature synthesis (SHS) in Ni-Al, Ni-Ti, Ti-Al systems for layerwise manufacturing of three-dimensional (3D) parts was suggested and experimentally realized for the first time.

Functionally Graded Structures (FGS) and FG objects fabricated by applying complex and dissimilar materials ensure specific properties of final product. The manufacturing of 3D FG objects by 3D laser

¹ Shishkovsky I, e-mail: shiv@fian.smr.ru



cladding (LC) and/or Direct Metal Deposition (DMD) is one of the most promising techniques capable of meeting various industrial challenges [3, 4]. The possibility of controlling the hardness of graded structures from layer to layer by changing powder composition and introducing reinforcing intermetallic inclusions into superalloy matrix widens the range of possible applications of 3D parts in aerospace and nuclear industries.

In the present study, it is offered to perform FGS fabrication in a TiNi-Al system by layerwise increase of Al concentration from the substrate up to the surface during 3D LC. Due to very different melting temperatures in high-Al (i.e. rich in Al) and low-Al regions, the identification of phase structures in the whole compositional range of TiNi-Al up to 1000 °C would be a very interesting task [4-7]. Optical microscopy (OM) and scanning electron microscopy (SEM), X-ray phase analysis and microhardness measurement were employed in this study in order to analyze the FGS.

2. Experimental procedure

The following materials were used for the FGS fabrication: aluminum powder with 99 wt% of Al and nitinol powder both produced by TLS Technik GmbH & Co (Germany) containing 99.76 wt% of intermetallic NiTi phase. The NiTi powder was represented by a 60 μm fraction. The pre-alloyed NiTi phase was obtained by means of gas phase atomization. The NiTi stoichiometric ratio was 45 wt% of Ti and 55 wt% of Ni. The Al powder particles were mainly spherical with the size of ~60-80 μm for 95% of them.

The substrates made of Ti-6Al-4V were round plates with the diameter of 65mm and the height of 5 mm. The powder size distribution was studied by means of a granulomorphometer ALPAGA 500NANO (OCCHIO s.a.).

All the experiments were carried out in the DIPI laboratory (ENISE, France) with the aid of the HAAS 2006D (Nd³⁺:YAG, 4000 W, cw) with laser beam delivery system, powder feeding system, coaxial nozzle, and digitally controlled 5-axes table [4]. Some features of the equipment are reported below:

- Powder feeder: 2-channel MEDICOAT, the powder feeding rate can be adjusted separately for each channel. Argon and nitrogen are used as carrying gases.
- Coaxial nozzle: the advantages of the coaxial injection are small heat affected zone (HAZ) and possibility of multidirectional cladding due to radial symmetry between the laser beam and powder flux. The shielding gases (Ar, N₂) are surrounding, focusing and protecting the powder flow and the melting pool from oxidation or, vice versa, causing the nitridation initiation.
- CNC center LASMA 1054 is applied for the displacement of the sample and nozzle relative to each other with the positioning accuracy up to 1 μm .

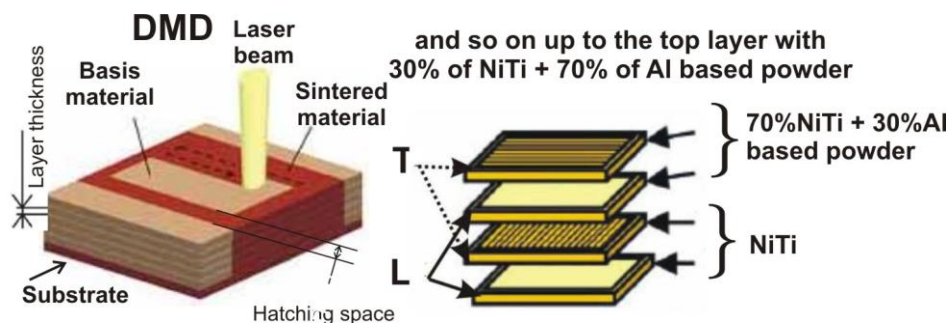


Figure 1. Schematic of the multi component graded structure fabrication by 3D laser cladding.
Longitudinal – L; transversal – T.

The method of the functional graded structures (FGS) fabrication used in the present study is schematically presented in Figure 1, as it was proposed earlier [4]. The hatching distance was 2 mm, the layer depth was ~2 mm and the powder feeding rate was ~10 g/min. The layers were made of NiTi

and Al powders on a steel substrate with the following strategy: the first two layers were of pure NiTi, the two next consisted of 70% NiTi + 30% Al-powder, the third couple of two equal layers – 50% NiTi + 50% Al-powder and at last, the two upper layers, 7th and 8th, had the ratio of 30% of NiTi + 70% of Al-powder. Each second layer was formed on the bottom layer after turning it by 90 degrees. Argon was the carrying gas. The laser scanning speed V_L was 400–600 mm/min, laser power P varied within the range of 800–1200 W and laser beam (LB) spot diameter d_L was 3 mm. The first channel of the feeder with NiTi powder had the gas flow rate of approximately 20 L/min, while the second one with Al powder, ~10 L/min.

The macrostructure of 3D samples was examined with the Neophot 30M optical microscope with digital camera and PMT-3M (OKB SPECTR Ltd., St. Petersburg, Russia) microhardness tester. The morphology of the laser clad layers was studied with the use of LEO-1450 scanning electron microscope (Carl Zeiss Microscopy GmbH, Jena, Germany) equipped with an energy-dispersive x-ray analyzer (INCA Energy 300, Oxford Instruments Analytical, High Wycombe, UK).

3. Results and discussion

In our opinion, the choice of metal used as the matrix material, the type and proportion of the component added to synthesize the intermetallic phase are fundamentally important for LC in Ni-Ti-Al system. Since laser irradiation (LI) is well reflected by aluminum, the more refractory nitinol could be used as the matrix material, and in accordance with the Ni-Ti-Al phase diagram, it is reasonable to expect the emergence of new stable intermetallide phases of aluminides, including γ Ni_x(Al,Ti) phases. The optical metallography (OM) is presented in Figure 2. The bottom clad layers are significantly different in terms of structure and chemical composition as compared to the top layers. The bottom layers have dendrite structure with clearly visible laser passages. Some porosity can be noticed in the sample volume (Figure 2, bottom). The top layers are the mixture of globular inclusions of different nature. Initial grey nitinol matrix was locally preserved. At last, the middle layers have a complex intermetallic structure, which we expected to use for accounting real strengthening due to the formation of the Ni_x(Al,Ti) phases. Boundaries between layers are clearly visible, especially in place of Al content change.

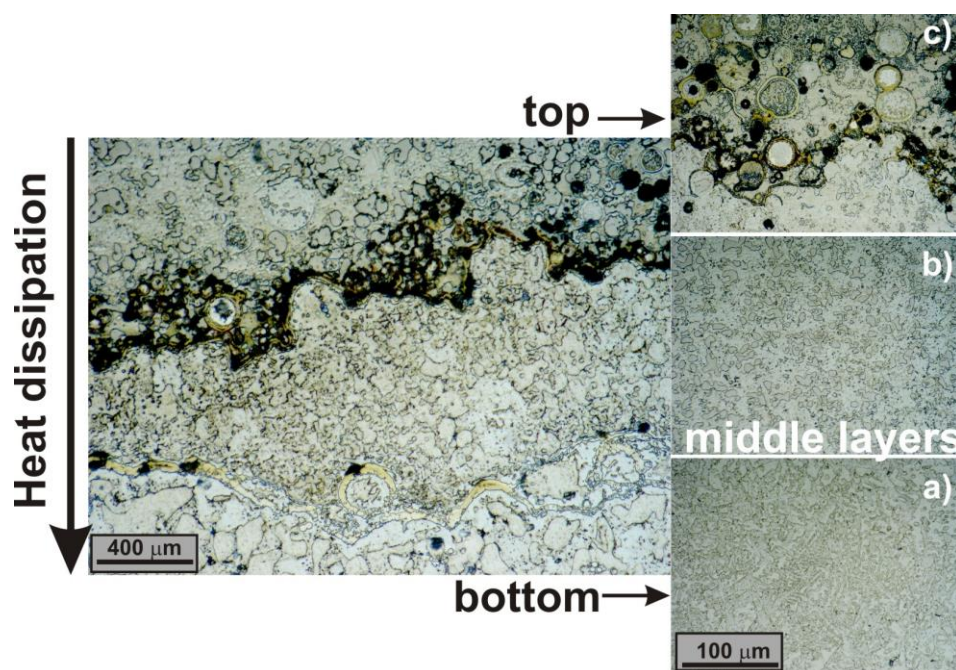


Figure 2. OM micrographs showing typical macro and microstructures of 3D laser clad coating of NiTi-Al multilayer system

The results of microhardness testing are shown in Figure 3. In the vicinity of the substrate, the microhardness is equal to 500-550 HV_{0.1}. With increasing distance from the substrate, the hardness increases and reaches 800-1000 HV_{0.1}. We connect the separate dips in microhardness in the bottom layers (up to 2 mm from the substrate, Fig. 3) with excessive porosity, and in the top layers, with the indenter entry into the complex Ni_x(Al,Ti) intermetallic phases. On the whole, the measured microhardness values significantly exceed other researchers' data (500-550 HV_{0.1} for the SLM of the NiTi by [8]) and could be estimated as a promising result of our study.

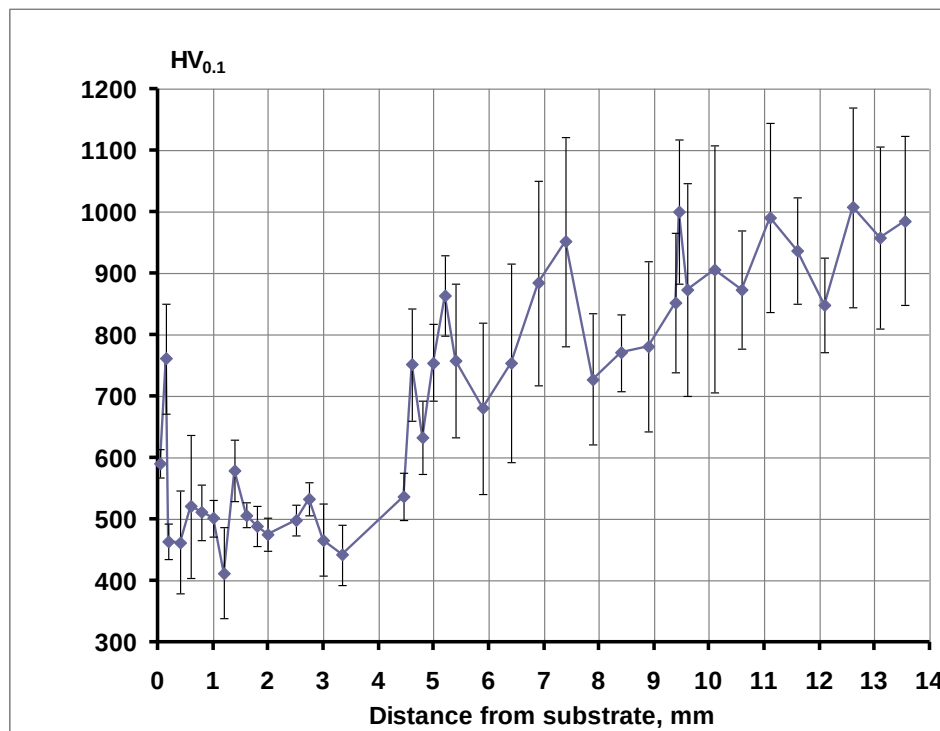


Figure 3. Microhardness of NiTi-Al FG multilayer system. Influence of distance from the substrate.

The SEM images of the micro- and substructures are shown in Figure 4 and enable more thorough study of microstructures appearing during the FG multilayer fabrication. The SEM images correspond to the bottom, middle and top layers of the FGS after the layerwise LC in the Ni-Ti-Al system. The top series in Figure 4 represent the SEM images of substructures under higher magnification than those in bottom series of the figure. It is evident that these substructures (Figures 4a and 4b) have dendrite nature.

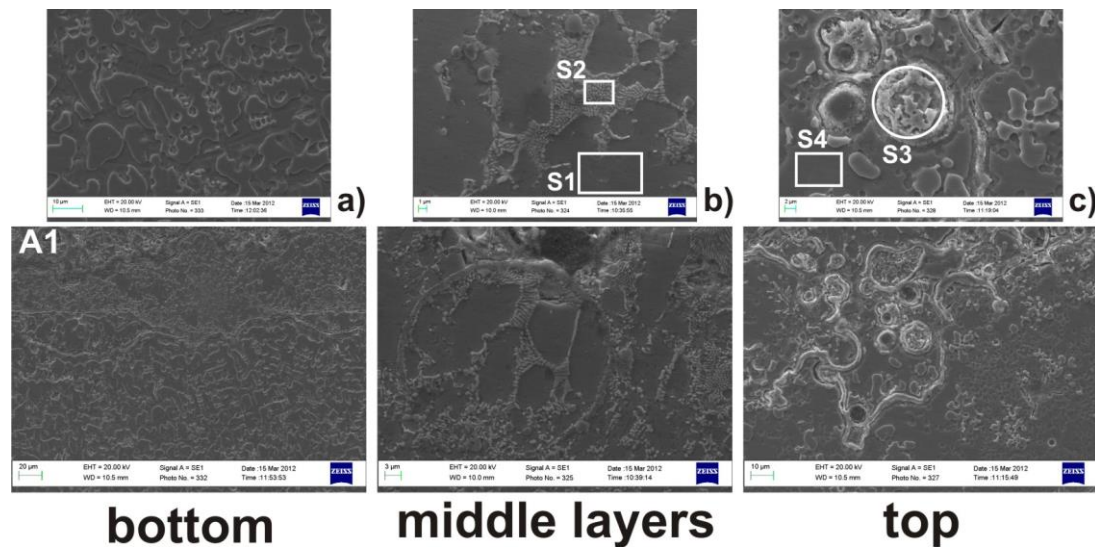


Figure 4. SEM micrographs showing typical solidification microstructures of bottom a), middle b), and top c) clad coatings with EDX results of NiTi-Al multilayer system.

The elemental analysis of regions S1-S2, S4 and A1 and the centesimal analysis of Ni-Ti-Al content also testify that we deal with the formation of a complex intermetallic phase. $\text{Ti}_{41.22}\text{Ni}_{38}\text{Al}_{20.78}$, $\text{Ti}_{27.37}\text{Ni}_{46.63}\text{Al}_{26}$ and $\text{Ti}_{39.99}\text{Ni}_{35.63}\text{Al}_{24.38}$ complex intermetallic phases, prepared by laser layerwise cladding, mainly consist of TiNi intermetallic compound exhibiting high hardness. Jung et al [9] considered that the precipitation of Ni_2TiAl Heusler phase with $L2_1$ -structure in a supersaturated B2-TiNi matrix and significant strengthening could be observed for NiTi-Al system under high temperatures.

Table 1. EDX data by Figure 4

Element, wt%	Ni K	Ti K	Al K	O K	Total
S4 area	38.00	41.22	20.78	--	100.0
S3 circle	36.22	44.23	9.37	10.17	100.0
S2 area	46.63	27.37	26.00	--	100.0
S1 area	35.63	39.99	24.38	--	100.0
A1 all area	46.43	53.57	--	--	100.0

4. Conclusion

The applicability of 3D laser cladding for creating a functional gradient and building of $\text{Ti}_x\text{Ni}_y\text{Al}_z$ intermetallic structures was experimentally studied. It was shown that under layerwise LC, the formation of a $\text{Ni}_3(\text{Al,Ti})$ intermetallic compound is observed in a NiTi-Al system.

Two-fold increase of microhardness from 500 HV to 1000 HV was obtained for similar samples due to the change of the element relation in the NiTi-Al system after 3D laser cladding.

Hence, the possibility of controlling the hardness of a multi-layer structure by changing powder composition was shown. Appropriate aluminum doping into nickel titanium can expand the range of 3D FGM application in aerospace or nuclear industries.

Acknowledgments

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