FP6 Project: NMP2-CT-2003-505954

RAMATI

RAPID MANUFACTURING OF TITANIUM IMPLANTS

Instrument: STREP

Thematic Priority: NMP

FINAL PUBLISHABLE REPORT

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Project coordinator organisation name INOV
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Introduction

Due to their complex shape, high dimensional accuracy, good surface finish, and one-of-a-kind production, dental prosthesis are usually manufactured by investment casting. Therefore, the materials used for this application must exhibit, not only excellent biocompatibility, ductility, toughness and wear and corrosion resistance, but also excellent castability, low solidification shrinkage, low reactivity with the mould material and the atmosphere, and high machinability.

The use of laser freeform manufacturing in prosthesis production will be a major breakthrough in many medical areas, and the aim of this project was to contribute to make it a reality. LFFM is particularly adequate for prosthesis production, since it eliminates most of the shortcomings of existing casting processes: (a) being a single-step process that produces near-net shape parts, the time expenditure and cost associated with multiple step fabrication are largely eliminated; (b) no machining is required; (c) higher material purity is obtained because contamination by tools, machining oils, lubricants, cleaning agents and binders is avoided and part manipulation is reduced to a minimum; (d) no tooling or fixturing is required; (e) manufacturing planning is automatic and easy; (f) the factory footprint, capital investment and human resources required are low; and, more importantly; (g) a turnaround time of a few hours or days instead of a few weeks for conventional processes. It is also an essentially waste-free process because unused powder can be reused, a feature that makes it attractive when expensive materials are used, as it is the case in biomedical applications

The RAMATI project aimed at establishing the scientific and technological basis for the application of Laser Freeform Manufacturing to the production of biomedical implants. The main applications targeted concern small size, customised prosthesis, for dental, maxilo-facial and cranial reconstruction applications, in general manufactured in a one-per-one basis, in order to allow exact tailoring of the prosthesis to the clinical requirements. The process should be fast and low cost, too.

The technology proposed in the present project is based on a seamless integration of advanced imaging techniques, such as TAC, computer-aided-design, and computer-aided-manufacturing, and advanced materials. These advanced technologies are use to create a complete solution for the production of high-quality, one-of-a-kind prosthesis, specially customised for individual patients.

Project objectives

The aim of the RAMATI Project was to develop **Laser Powder Deposition as a technique for the rapid production of small size customised titanium implants and prosthesis for dental and orthopaedic reconstruction applications** .

The main output of the project was expected to be a low cost “turnkey” Laser Freeform prototype system, manufacturing procedures and new materials for rapid manufacturing of customized titanium implants and prosthesis.

Laser freeform manufacturing should overcome the shortcomings of conventional processing technique, in particular by avoiding the need for a multitude of processing technologies, complicated work piece handling circuits and long manufacturing time. It should also allow for reduction of the processing costs, parts inventory, materials consumption and waste.

Hence, the RAMATI **specific scientific and technical objectives** are the following:

(a) Objectives related to the process equipment development

- To develop design and process control software;
- To develop a specific LFFM equipment;
- To integrate hardware and software in a LFFM equipment for implant production;
- To optimise the machine for dental, cranial and maxillo-facial implant production;

- To consider a possible extension of the process to other applications, such as orthopaedics.

(b) Objectives related to the process associated materials development

- To develop new materials fulfilling both the requirements associated to the laser powder deposition process and the expected biomedical application;
- To optimise these materials for dental, cranial and maxillo-facial implants;
- To consider possible extension of the application of these materials to other biomedical areas.

RAMATI was expected to bring the following main **innovations** "beyond conventional approaches":

• **Integration of advanced imaging techniques and materials**.
• Manufacturing based on **file transmission/ remote controlled manufacturing**, fully exploiting the possibilities created by the web
• **Low cost and easy to use equipment** allowing to manufacture **prosthesis "close to the patient"**
• **Special designed materials**
• **Combinatorial laser-assisted methods applied to the design of new biomaterials conceived for the RAMATI application**
• An **innovative concept and design of a low cost laser freeform manufacturing machine**

Partnership :

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FOTONA d.d – Slovenia
Faculdade de Medicina Dentária de Universidade de Lisboa – Portugal
Instituto Superior Tecnico de Lisboa – Portugal
ALMA Consulting Group – France
Microbotic - Denmark

Work performed and results achieved:

Equipment

Three different equipments were set up at INOV, FORCE and IREPA with three different laser sources.

Force has worked with 2 different laboratory set-ups and has in addition been responsible for the final integration of the demonstration prototype. The first two laboratory set-ups were based on an existing off-axis powder nozzle and powder delivery system capable of delivering powder rates down to roughly 1.5 g/min. An existing maximum 4 kW Nd-YAG-laser source was also used, albeit at a low power typical in the range 150-200W. The system is shown on Figure 1.

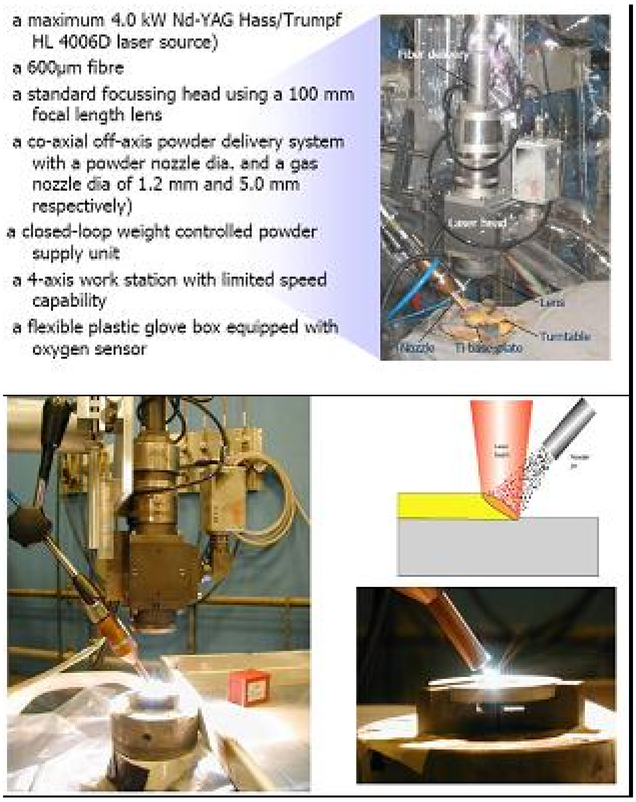


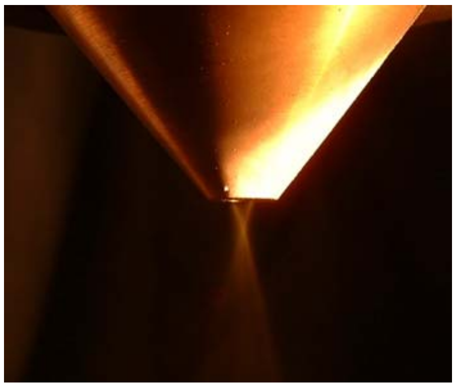
Figure 1. The 2nd generation laboratory set-up at Force.

Process development:

Nozzle study and setup

In the first part of the project, the study of the nozzle has been carried out with the objective to permit the manufacturing of thin walls, made of titanium alloy, in order to be able to build thin structures like dental implants.

IREPA LASER has developed the concept of the coaxial nozzle, first for diode laser and then for a higher beam quality due to the problems encountered with the diode lasers systems. Thanks to several numerical modelling, the powder stream (powder+gas) has been simulated, and allowed to define the size and shape of the internal cones of the nozzle. A prototype was designed and optimized for the high beam quality laser. The minimum effective merging point diameter of powders was measured down to 0,3 -0,4 mm, and the deposition efficiency up to 50%. According to these results, 2 new different nozzles were developed for 2 other different lasers, to be used by the partners of the project.



The powder stream and the nozzle

IST tested a conventional coaxial nozzle adapted to the CO2 laser, and worked on a original and new off-axis and microscale version, in order to evaluate the interest of such concept.

LFFM tests

LFFM tests have been performed to test system integration and performance. Straight walls and cylinder test samples were made by LFFM using a Co alloy and Ti6Al4V powders. Samples have wall thickness below 0.4 mm, but for improved dimensional accuracy and surface finish it will be necessary to use a more confined powder jet. On the other hand, wall thickness increases during build up due to heat accumulation, revealing the need for on-line temperature control.

A micro scale deposition system whereby powder particles are injected via capillary normal to the substrate was installed and evaluated. LFFM tests have been performed with the new deposition system using grade 2 Ti powder. A preliminary investigation into the effect of process variables and parameters was conducted. Single tracks and thin-wall structures were deposited. An assessment of process parameters on powder catchment efficiency for single tracks was conducted along with assessments of track geometry in the form of track dimensions, alpha angle and cross-sectional area. The effect of process parameters as a means of control of thin wall geometry was evaluated.

An on-line control system was developed whereby the melt pool geometry is maintained constant during deposition by varying laser beam power with proportion to changes in the radiation emitted by the melt pool in the wavelength range (1-1.7 µm) as monitored by a photodiode. A PID type control function was applied to the deposition of thin walls resulting in walls with constant cross sectional width and a relatively low surface roughness.

A similar development has been carried out using the coaxial nozzle, previously presented. The first tests with a diode laser demonstrated that the bad quality of the beam didn't allow a full melting of the powders. The process has been then implemented with different lasers, and the tests showed the need of a high beam quality in order to obtain a full melting of the powders with flexible operating conditions. With these new parameters, 0.5 mm width walls with a power of 100W were achieved, with a stable interaction. A process window has been established. The width of the clad is essentially linked to the volume of powders which are melted within the beam. This volume depends on laser power, power density, beam geometry and powders characteristics and geometry. Thus, over a large range of parameters, the width of the clad is nearly constant. On the other hand the power has a direct influence on the clad width. In the range of 500 up to 1000µm, the width can be adjusted by using an adapted power, while a sweeping of the beam is needed to adjust the wall width, when the wall width is larger than 1mm. The tests have also showed that a low powder feed rate is needed (<200mg/min) to improve the process conditions. However, it has been showed that the main limit of this process is the closure of volumes, due to collision between the cones of the nozzle and the part to be manufactured, particularly when the top surface is concave. Moreover, no specific software exists, which could take the process constraints into account.

LFFM sample characterization

In these tasks detailed characterization of the defects, microstructure, hardness and mechanical properties of samples produced by LFFM was performed.

Several LFFM samples produced by different partners using Ti gr.2, Ti6Al4V and Co-Cr powders were tested. Evaluation of the defects (presence of pores and cracks) was performed by X-ray analysis of the walls. A detailed structural characterization was made by optical microscopy, scanning electron microscopy, chemical composition determined by EDS microanalysis, phase identification was accomplished by X-ray diffraction. The hardness of the samples was evaluated by Knoop microhardness tests, performed normal and parallel to the deposition direction. The mechanical properties of the samples were evaluated by tensile tests and three-point bending tests.

The microstructure and properties of the LFFM samples were compared with those of benchmarking materials used in dental applications produced by conventional techniques in WP7.

Software Development

Within the framework of RAMATI, two software applications were developed.

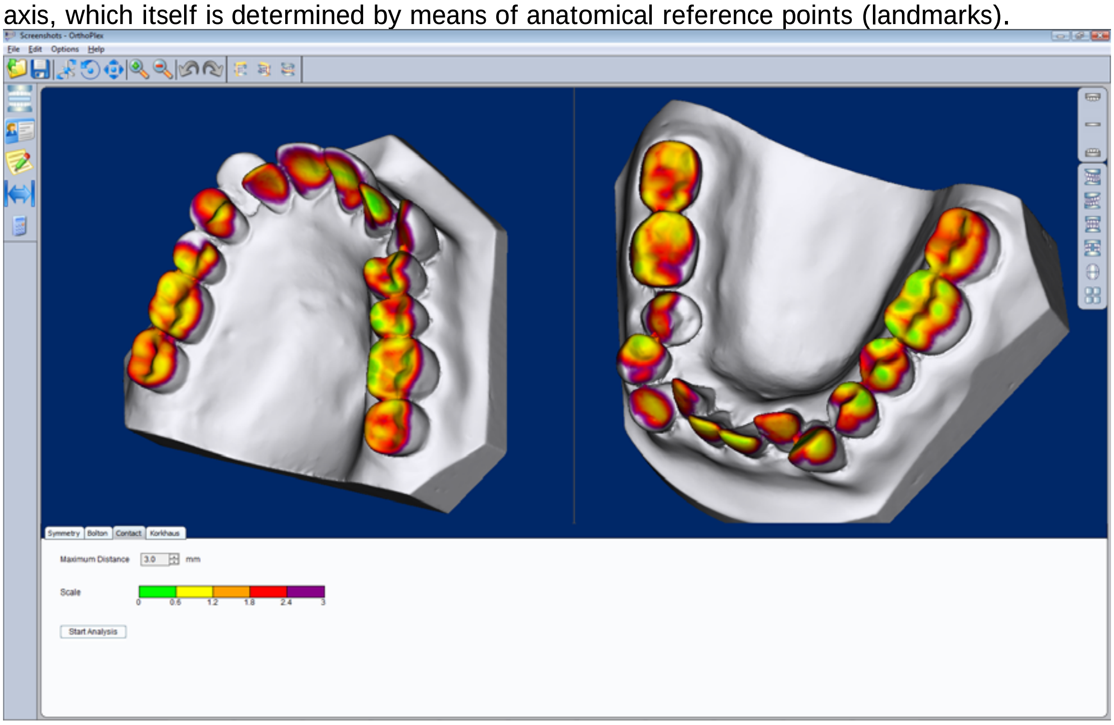
The first application is a viewer for evaluating and analyzing digitized dentitions in preparation of orthodontic treatment. This application is regarded as being illustrative for software in the dental field relative to the requirements for representing and manipulating complex dental geometries. The viewer relies on discrete surface representations to visualize the dental structures on screen. Since its main functionality consists of viewing of and measuring on digitized dentitions, the user interface was designed with special care. The objective was to create a 3D environment with which dentists would be as comfortable working as with physical plaster models. Therefore it was designed in close collaboration with clinical advisors. Aside from basic functions as rotating, zooming and panning the program also allows dentists to measure tooth widths, tooth heights, overjet, overbite, angles and other parameters relevant to orthodontics. The measurement values are used to run different diagnostic analyses from within the application:

- Bolton analysis:
- Korkhaus analysis
- Symmetry analysis

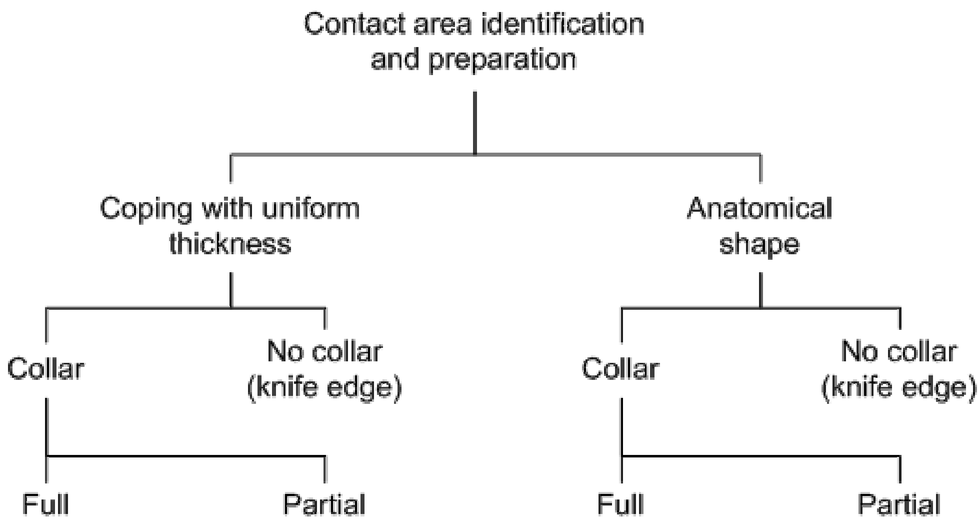
The user interface of the application also allows clipping of the digital tooth models. This feature is important to accurately assess the points of contact between individual teeth in the upper and low jaw. Another feature in the software is an occlusogram, which displays by means of a color code, the distances between opposing teeth in opposing jaws. Comparing occlusograms of pre-and post-treatment models allows the user to analyze effectiveness of the treatment.

Plans for technological implementation of this first application include the commercial launch of the system together with a leading manufacturer of orthodontic equipment. Therefore additional developments have been conducted (outside the scope of the RAMATI project) to allow online file handling and online ordering. In addition an advanced scanner interface has been created to assure semi-automated digitization of the intra-oral anatomy.

The servicing version of the software (i.e. the Master version) comes with a "preparation" module for the purpose of generating viewer projects. This module incorporates functions intended for cleaning and editing scan data to obtain digital impressions with a finishing equivalent to that of traditional plaster casts. Moreover registration and alignment tools have been built in to allow correct positioning of the digitized jaws relative to each other. Jaw orientation is defined relative to the intercondylar axis, which itself is determined by means of anatomical reference points (landmarks).



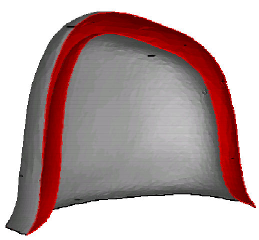
The second application developed within RAMATI is a dental design module for generating and editing new prosthetic structures based on anatomical data described by means of discrete (triangle based) surface representations. More in particular the application is intended to design dental crowns and copings based on tooth stump information acquired from the patient. The developments were performed using MDCK (Materialise Digital CAD Kernel) technology with an existing software front-end (3Matic, Materialise). All software routines were integrated into a "dental" plug-in. To run the application, the receiver site (i.e. the tooth stump) must firstly be digitized and a 3 dimensional model of the anatomy must be generated. Though this initial step was not under investigation in the framework of the RAMATI project some dedicated routines were created to allow point cloud editing, noise reduction and meshing of structures digitized with dedicated commercially available dental scanners.

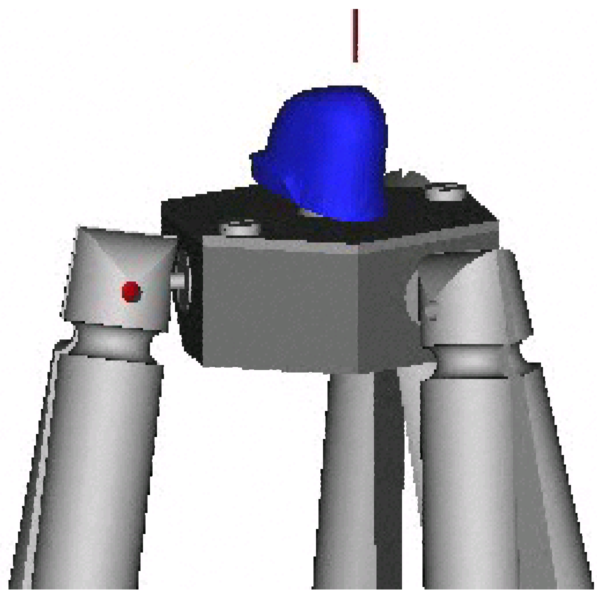


Once the input data is available the user can choose to design one of different kinds of copings or crowns. Depending on the nature of the design, more or less complex routines are used to create a proposal. The two most determining factors are (i) the need to have an anatomically shaped part or not and (ii) to need for a collar or not.

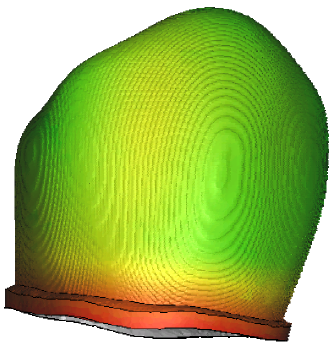
For designing crowns and copings with anatomical shape, the system currently also requires the digitization of a dental wax-up prepared by the dental lab, indicating the desired shape of the tooth restoration based on simulations in an articulator. The surface representation of the wax-up can be digitally matched to that of the tooth stump by means of registration algorithms available in the plug-in. Once done, the wax-up is indicative of the “boundaries” within which the coping must be designed in order to achieve both an aesthetical and functional result. In the future the wax-up may be generated directly by the software using teeth form a digital tooth library.

Collars can be added to the designs to reinforce the copings where necessary. They follow either the full circumference of the tooth stump or only one (typically the lingual side of the restoration). If no collar is required, the coping is designed with a knife-edge finish. The figure below shows an example of a typical design in section.





In addition to design functionality the application also includes post-processing tools required for the creation of production files that can be used to manufacture the parts physically. Three routines can be identified: midplane calculation; auto-positioning and thickness calculation.
Because the cladding tracks (also in RAMATI) are relatively broad, thin walled structures such as copings must be built up with a single continuous clad of the laser. This means that the coping design must be reduced to a single surface to which thickness information can be assigned depending on the position in the 3D space. For this purpose Materialise has created software functionality allowing automatic determination and creation of the middle plane in between the inner and the outer surface of the coping design. A positioning routine further orients the coping in a specific manner relative to the center of the build platform of the hexapod onto which the part will be constructed. Thickness information can be added to each discrete point on the surface of the part by means of color code. This information can afterwards be used during production to vary the power of the laser or the speed along the path in order to obtain the required thicknesses.



Material development

Titanium has been selected as the raw material to be used for this process.
In the first task of WP2, which is the development of the process, the spherical Ti powder is produced by the Plasma Atomisation process. This process ensures the low oxygen content and the very good sphericity of the powder. Pyrogenesis was in charge of production of powder and worked on the optimisation of the process. Pyrogenesis identified the process parameter in order to produce Ti powder with the desired characteristics. Then, the objective was to try and increase the productivity of the system, thus producing more powder per batch, without altering significantly the desired properties and characteristics of the powder.
A major achievement is the increase in the productivity of the system (~ 20%). Moreover, the oxygen content in the powder has been controlled again in the desired levels (<0.15%), due to the addition of Ar in the preheating system. Another improvement has been observed in the particle size distribution, ~ 90% of the mass of the powder is considered fine particles (diameter < 75 µm).

WP5 was devoted to Biocompatible Raw Materials Development. In particular, tasks 5.1 dealt with the knowledge based design of materials for prosthetic applications. During this task manufacturing and characterisation of NiTi alloy was carried out. SHS (Self Propagation High Temperature Synthesis) technology was used for the synthesis of shape memory alloys (NiTi). This technology allows the synthesis of highly novel material with low energy consumption and high purity products are obtained. The best synthesis conditions were optimised for NiTi powder manufacturing. A complete microstructure characterisation of the product was carried out.

Variable powder feed-rate laser cladding was used as a combinatorial materials synthesis technique to produce composition gradient (Ti-Mo, Ti-Zr, Ti-Ta and Ti-Mo-Zr) materials along a clad track, which

were screened in order to find individual compositions with promising combinations of properties. The microstructure, mechanical properties and wear and corrosion resistance of these alloys were investigated. Some interesting composition ranges were identified for each alloy system for further study using constant composition coating, according to appropriate criteria. Compositions with great potential for orthopaedic applications (low Young's modulus) and for dental applications (higher values of Young's modulus) were selected.
Samples of the Ti-X alloys selected and characterized in detail in terms of microstructure, mechanical and wear properties and compared with the benchmarking materials produced by conventional techniques. These samples were also submitted to biocompatibility tests in WP8.

Modelling
A finite element of LFFM model in development at IST has been adapted to LFFM of Ti and Ti alloys. The model allows predicting the influence of laser processing parameters on the phase transformations that occur on cooling the material during and after LFFM. These affect the materials final microstructure and its properties. A literature search for Ti and Ti alloy physical constants has been carried out and used as input parameters for the model.
The model was built for grade 2 cp-Ti and the accuracy of the results tested against experimental results for laser melting of titanium substrates. The model to simulate phase transformations during laser powder deposition was adapted to the deposition of Ti-6Al-4V and used to study the influence of substrate temperature on the final microstructure of walls fabricated by LFFM. The model was used to simulate heat transfer and phase transformations that occur during laser free-form manufacturing of a titanium dental crown.

Benchmarking activities

Comparison with conventional materials and emerging technologies
For the purpose of technology validation and benchmarking analysis, samples processed using conventional technologies (plastic deformation and casting) and by the Selective Laser Melting (SLM) process, another emerging technology, were produced and characterized. A detailed characterization of the defects, microstructure, hardness and mechanical properties of samples was performed. Their microstructure and mechanical properties were than compared with those of LFFM samples tested in WP2.
The following samples were tested:
- Wrought Ti Gr.2
- Cast Ti Gr.2 samples
- Ti Gr.2 SLM samples
- Ti6Al7Nb and Ti6Al4V SLM samples

Microstructural characterization by optical microscopy, SEM and XRD was performed and the mechanical properties assessed by microhardness, ultramicroindentation, tensile and 3-point bending tests.
It was shown that SLM samples are more fragile and less ductile than the same materials produced by conventional and LFFM methods. This behaviour is due to presence of a significant amount of porosity in SLM samples that result from lack of melting of the powder. On the contrary, the comparison shows that LFFM samples have properties very similar to those of the materials produced by conventional techniques and can be used in the same type of application.

WP8 was dedicated to the assessment of the biocompatibility of the biomaterials developed during the project. Different Ti alloys and processes were evaluated, such as NiTi, TiZr, Ti6Al4V Ti6Al7Nb and Ti pure. Ti Grade II and AISI 316L were used as controls of commercially available materials.

Biocompatibility evaluation of the samples was carried out focussing on "ISO 10993-1: Biological evaluation of medical devices" and "ISO 7405- Dentistry- preclinical evaluation of biocompatibility of medical devices used in dentistry- test methods for dental materials". Ion Release assays were carried out based on "ISO 10271: Dental metallic materials- Corrosion test methods" .Due to lack of possible interpretation of test results in this standard, results were compared with limits related to maximum daily intake and upper tolerable levels.

The analysis did not see any negative issue concerning biocompatibility of the studied samples, (as far as processing did not add pollutants) and all the ion release values of the samples were reasonably below the limits, with NiTi showing the highest ion release levels by far. Therefore, NiTi should be considered in a case by case basis.

On the other hand, a review was carried out to identify the European Standards and Directives related to medical devices and dentistry, the Corrosion Tests in dentistry and a literature survey of Ti alloys used for medical applications, in order to select possible compositions of Titanium alloys.

Integration

The final integration of the demonstration prototype (WP6) was performed at Force. In this third generation system a more advanced setup were used integrating the obtained knowledge and the combined effort of all partners. It was during the technical work of the project realised that a laser source of very high beam quality and a maximum power exceeding roughly 120W was needed in order to be integrated with the co-axial powder nozzle, and the only laser sources types capable of fulfilling these demands were either a fibre-laser or a disk laser source. A new powder nozzle was developed by IREPA for the 280 mm focusing laser optic used, and the nozzle was tested in the new test setup fulfilling the expectations without major problems. No suitable powder feeding system was however available for this third generation system and therefore Force developed a new powder feeding system. In principle it is based upon a standard bowl feeder that has been stabilised through a special feed back controlling system, and this powder feeding system is able to deliver very small, but still very stable powder flows down to 100 mg/min. The prototype also included the hexapod microrobot from Microbotic and the controller of the hexapod robot was integrated to the controlling system of the new fibre laser, so that the laser was controlled through the robot controller.

The demonstration prototype thus was composed of:

- A 200 W leased fibre laser source from IPG
- The powder nozzle developed by IREPA
- The new powder feeding system developed by Force
- The hexapod Robot manipulator developed by Microbotics
- A purpose built optical head
- A glove box enclosure

Initial robot programs were developed for building up simple geometries in the form of linear walls, cylinders and cones with the aim to carry out initial test of the laser process and to test the different equipment integrated in the setup. Programming of the hexapod robot through CAD drawings of relevant tooth cap geometries has been carried out by Microbotic based on the files from Materialise. Despite some limitations in the robot speed and acceleration performance the laser process itself showed very good possibilities for building up the relevant complex geometries for e.g. dental caps. A demonstration of the prototype was made during the final meeting at Force in June 2007. The demonstration prototype itself and while processing is shown on figure 2.

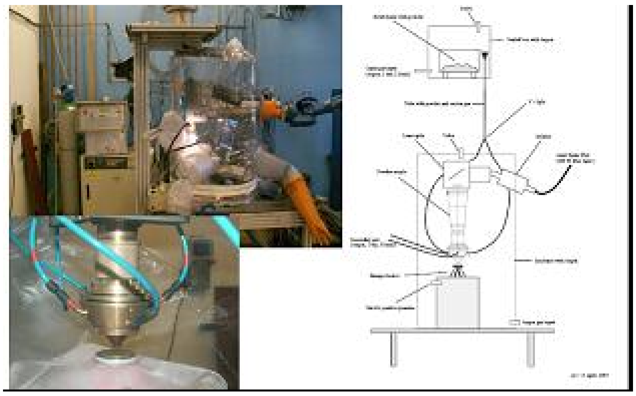


Figure 2. The final prototype (left) and while processing (right).

A second prototype was built at RTM and tested at IREPA.

RTM built compact but fully specified LFFM system (see figure 3), which can be equipped in a modular architecture on the basis of different requests. The system has versatility to accept different laser types to be used for special product to be manufactured.
The prototype system was transferred to IREPA for integration of the source (fiber laser) and the implementation of the LFFM accessories, including the powder feeding nozzle.

Fig 3. - Complete machine ready for installation and testing

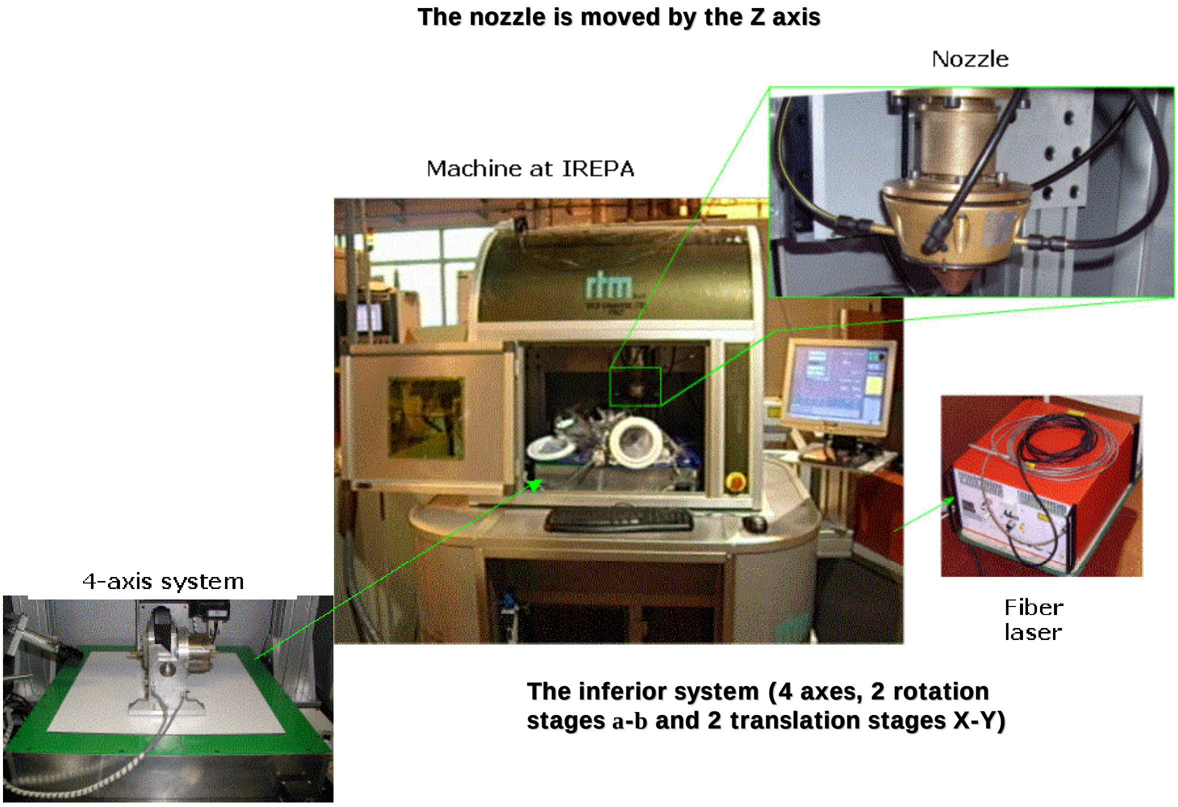


Fig. 4 –Integration of the machine at IREPA

In a first step, the process has been set up with this new equipment and the process was validated on simple geometry.

In a second step, 5 axis manufacturing was developed in order to evaluate the limits and the constraints of this process, and to demonstrate the feasibility of 3D objects. Figure 5 shows a significant example of a programmed 3D form.

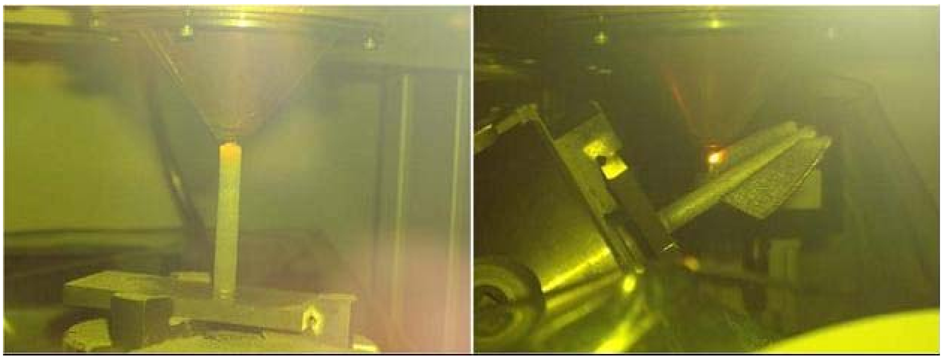


Fig. 5 — LFFM of a cylindrical form (left) with helical wings (right)

The machine was validated from the process point of view, making simple 3D shapes. The feasibility of the 5 axis manufacturing has been demonstrated. Moreover this work allowed to show the difficulties of the 3D building in the case of a simple part, mainly concerning the start and stop of the cycle, and the control of the deposited thickness.

Conclusion

1. The RAMATI project was successfully concluded and lead to significant developments in what concerns the scale-down of the LFFM process in order to be able to produce parts with dimensions in the 100 micron size scale.
2. A new coaxial deposition nozzle for microdeposition was developed.
3. New powder feeding concepts for microdeposition were developed.
4. A new powder feeder design was developed,
5. A new software allowing the manipulation of CAT and scanner data and the design of dental prosthesis and implants on the basis of these data was developed and made available commercially.
6. Two new designs of microdeposition equipment were developed.
7. Deposition procedures for Ti-alloys dental prosthesis and implants were established.
8. A new synthesis method to prepare NiTi alloys was developed.
9. A new combinatorial method of alloy design was established.