TESTING TITANIUM IMPLANTS

Sorin Mihai CROITORU[1,*], Adrian Ion PACIOGA[2]

[1)] Assoc. Prof., PhD, Machines and Manufacturing Systems Dept., University “Politehnica” of Bucharest, Romania
[1)] Senior Researcher, National Institute for Research and Development in Mechatronics and Measurement Technique of Bucharest, Romania

Abstract: *Dental and orthopaedic implants are supposed to be tested from medical and/or engineering points of view. This paper presents some engineering experimental research the authors made, related to titanium implants produced by the Romanian manufacturer of dental implants, Tehnomed Impex Co S.A. and the National Institute for Research and Development in Mechatronics and Measurement Technique of Bucharest (orthopaedic implants). Part of the experimental research was performed in the framework of a research contract financed by Romanian Ministry of Research and Education. The tests related to torsion resistance after chemical treatment of the implant were performed in order to see if the implant has the same (or at least enough) resistance even if its wall thickness is diminished after chemical treatment and its surface’s micro geometry is modified. In this case the implants have enough resistance compared to the non treated implants, but the micro geometry has lower quality because of the chemical attack. Tests related to the crystallographic structure of the implant’s material compared the methods of obtaining the implant: rolled bar and rapid prototyping laser sintered bar. In this case the rapid prototyping laser sintered implant has a more compact crystallographic structure, but the cost is greater.*

Key words: *titanium implant, torsion, crystallographic structure, rolled bar, rapid prototyping.*
¶

1. INTRODUCTION

This paper presents the research on implants made by the Romanian manufacturer, Tehnomed Impex Co S.A. and the National Institute for Research and Development in Mechatronics and Measurement Technique. The research subjects were imposed by special chemical treatment of the implant surface or manufacture method.

In case the surface of the implant is chemically treated a thickness diminish of the implant’s wall is involved due to the chemical reaction. This thickness diminish could be as much as 0.06–0.1 mm. Dental implants made by Tehnomed Impex Co S.A. have small wall thickness, less than 0.5 mm, between the inner threaded hole, used for fixing the superior structure and external thread used for fixing into the jaw bone (Fig. 1).

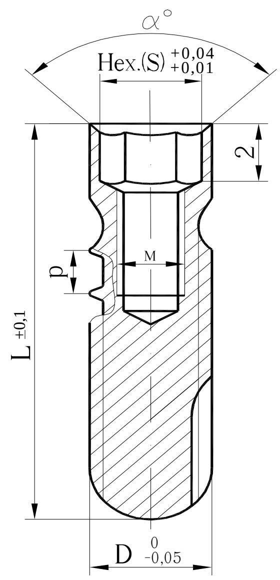

Fig. 1. Dental implant (Tehnomed).

During the insertion into the bone, the implant has to be screwed, thus implant torsion is involved. The following question appeared: will the implant cease to torsion during the insertion because the smaller wall thickness due to the chemical treatment? The answer to this question is given by the torsion test of the implants, both chemically treated or not.

Another question appeared because of the new titanium implants manufacturing method which is rapid prototyping. This method is used by the National Institute for Research and Development in Mechatronics and Measurement Technique of Bucharest to manufacture personalized orthopaedic implants. Because the material, in this case titanium, is layered point to point is the resulted material porous? In order to answer this question two samples of titanium, one sample of rolled bar and one sample obtained by rapid prototyping, were studied related to their crystallographic structure.

Both research subjects lead to interesting conclusions, but it should be mentioned the implants were very good in all cases, having enough strength to be used instead the bones they were supposed to replace. The only threats, other than medical ones were the imposed micro geometry and cost.

* Corresponding author: 313 Spl. Independenţei, Sector 6, 060042 Bucharest, Romania,
Tel.: +40214029174; mobile: +40722623858;
Fax: +40214029420.
E-mail addresses: *sorin.croitoru@gmail.com* (S.M. Croitoru); *pacioga@yahoo.com* (A. Pacioga)

2. TORSION TESTS OF DENTAL IMPLANTS

2.1. Preliminary research data

In the framework of a research project two series of dental implants prototypes ϕ4x12 mm were chemically processed. The difference between these two series was the implants' material, respectively pure titanium, grade IV and alloyed titanium TiAl6V4. Manufacturing and mechanical testing were done by Tehnomed Impex Co S.A. and POLITEHNICA University of Bucharest, Cutting and Cutting Tools Lab. of Machines and Manufacturing Systems Dept. Chemical processing of the dental implants was done by METAV-CD S.A.

In order to characterize the dental implants shape after the chemical processing, there were done measurements at the Cutting and Cutting Tools Lab. of Machines and Manufacturing Systems Dept. Measurements were done on the workshop microscope RK-3, having the accuracy of 0.001 mm (Fig. 2).

Evidently, measurements were done before and after the chemical processing.

Without giving the measurements results, some observations could be presented in the following.

In case of dental implants made of pure titanium, grade IV:

- head of the implant is no longer cylinder, but a barrel;
- generally, the dimensions diminish after chemical processing by 0.02 mm, in average, with a minimum of 0.008 mm and a maximum of 0.085 mm for different dimensions;
- chemical reaction is not uniform, leading to the shape modification of the implant;
- in one case, a chemical deposition was observed;
- comparing the sanded implants to the sanded and chemically processed implants a pregnant alveolar structure is observed in the first case. In the second case, the alveolar structure is lower, even disappeared in some areas.

In case of dental implants made of alloyed titanium TiAl6V4:

- one implant presents depositions;
- diminish of the dimensions is 0.007 mm minimum and 0.044 mm maximum;
- chemical reaction is not uniform, however the implant head looks more like a cylinder than a barrel, compared to the pure titanium implants;

Fig. 2. Workshop microscope RK-3.

- the last observation related to the sanded surface is the same as in case of dental implants made of pure titanium.

Because diminish of the dimensions of the chemically processed implants the question of implants' smaller resistance appeared. This is why some torsion tests of the implants were done, in order to their crack.

It should be mentioned that in addition to the chemically processed implants there were manufactured another 3 implants made of pure titanium and another 2 implants made of alloyed titanium. They were used as reference in torsion tests of the chemically processed implants.

a

b

Fig. 3. KISTLER dynamometer:
a – measuring element and non-contact receptor;
b – CPU and PC with specialized software Dynoware.

Fig. 4. Prototype implants, KISTLER dynamometer measuring element and used tools for insertion.

Fig. 5. Prototype implants, used tools and bone for insertion.

Measurement of the torsion moment was done in the Cutting Tools Lab. using a KISTLER dynamometer with the sensible element used for measuring drilling cutting forces: axial force and torsion moment. The dynamometer is presented in Fig. 3,*a* and *b*.

Tested implants and some of the used tools for insertion (tubular wrench and implant adaptor) are presented in figure 4.

Considering that the real danger for dental implant cracking occurs during its insertion into the bone, the following insertion was designed: the dental implant is inserted into a pig bone, by its torsion using a hexagonal adaptor, the negative hexagon of the dental implant.

The adaptor is driven by means of a tubular wrench, which is fixed into the collet of the KISTLER measuring element (see Fig. 3,*a* – tubular wrench fixed into the collet and Fig. 5 – set of tubular wrench).

In fact, the pig bone was driven (rotated) while the rest of the elements were fixed. The KISTLER measuring element sensed the torsion moment during this insertion operation.

In respect to ASTM F67-06, chemical composition of pure titanium and tensile resistance are presented in Table 1, for each grade.

From Table 1 it is revealed that with increase of the alloy elements percentage the tensile resistance increases. The same trend exists in case of alloyed titanium, tensile resistance reaching even 100–120 MPa. This means, practically, the tensile resistance of the alloyed titanium is twice the tensile resistance of the pure titanium.

2.2. Experimental results

The experiments objective was to reach the torsion moment for cracking chemically processed dental implants, in order to compare them to not chemically processed dental implants.

In the following, the succession of the experiments will be presented, together with the graphs of the torsion moment measured with KISTLER dynamometer.

It should be mentioned from the beginning the dynamometer has its own adjustments, the measured values being the real ones. However, some tests were made to verify the dynamometer functioning, for reasons that will be presented in the following.

First test was done by screwing the first reference dental implant made of pure titanium into a pig bone, in which were made holes of 3 mm diameter, unthreaded (see Fig. 5). The aim was to self threading the implant followed by its blocking and, eventually, cracking. It should be mentioned that in the real case the implant is screwed into a threaded hole.

Torsion moment is presented in Fig. 6. It was concluded that the torsion moment does not increase above 1.34 Nm because the adaptor deformed losing its hexagonal shape. It could be possible that the hexagonal shape of the adaptor was not accurate from the beginning (the adaptor could be used). Consequently, another new hexagon shape was cut; the experiment was remade obtaining the situation in Fig. 7.

Again, the hexagonal shape of the adaptor deformed.

Because the adaptor was made of alloyed titanium, it was considered that if the adaptor will be made of carbon steel for cutting tools it will resist. So, with the new adaptor made of carbon steel for cutting tools the measurement was the one presented in Fig. 8

In this last test the hexagonal adaptor deformed again. Furthermore, the pig bone cracked between the initial hole and its end. This shows, if necessary, the insertion must be done in a threaded hole made in the bone.

In these circumstances, the tests done on the reference 1 dental implant made of pure titanium was abandoned.

Table 1

Chemical composition of pure titanium grades (ASTM F67-06) in mass percentage and tensile resistance

Alloy Element	Grade 1	Grade 2	Grade 3	Grade 4
Nitrogen, % max	0.03	0.03	0.05	0.05
Carbon, % max	0.08	0.08	0.08	0.08
Hydrogen, % max	0.015	0.015	0.015	0.015
Iron, % max	0.20	0.30	0.30	0.50
Oxygen, % max	0.18	0.25	0.35	0.40
Titanium, %	rest to 100%	rest to 100%	rest to 100%	rest to 100%
R_m, MPa	240	345	450	550

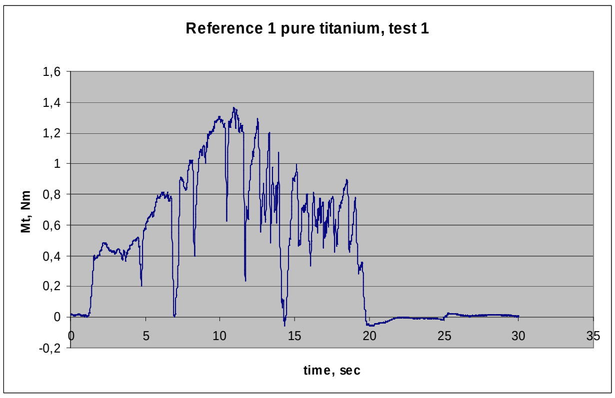

Fig. 6. Experimental torque, 1-st not treated implant, test 1.

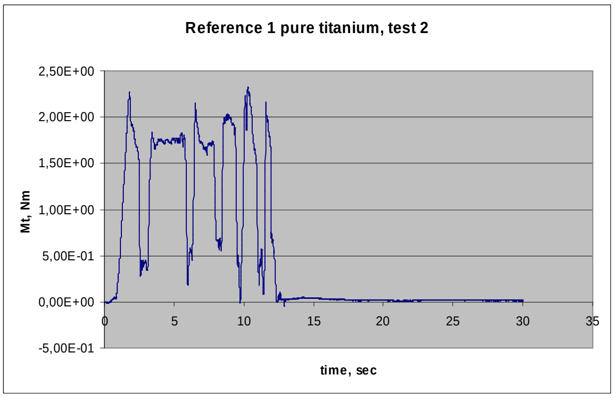

Fig. 7. Experimental torque, 1-st not treated implant, test 2.

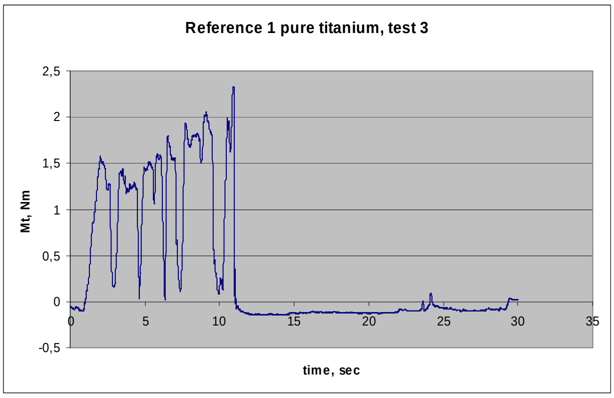

Fig. 8. Experimental torque, 1-st not treated implant, test 3.

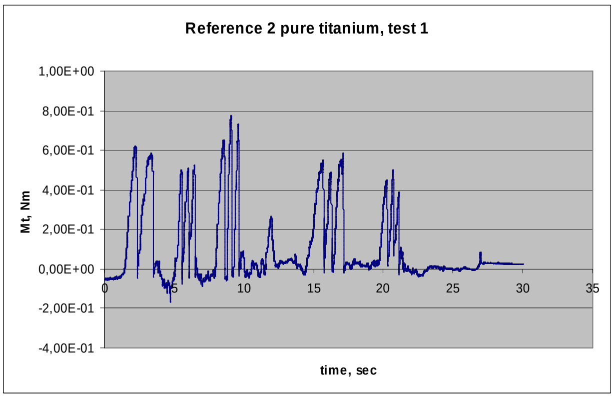

Fig. 9. Experimental torque, 2-nd not treated implant, test 1.

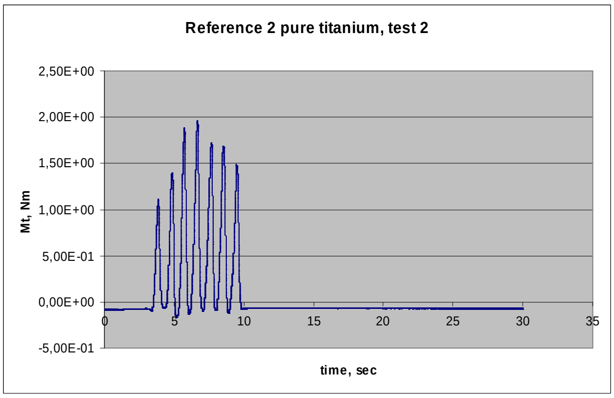

Fig. 10. Experimental torque, 2-nd not treated implant, test 2.

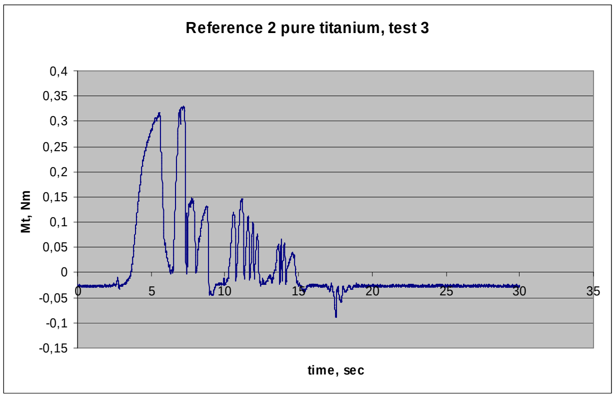

Fig. 11. Experimental torque, 2-nd not treated implant, test 3.

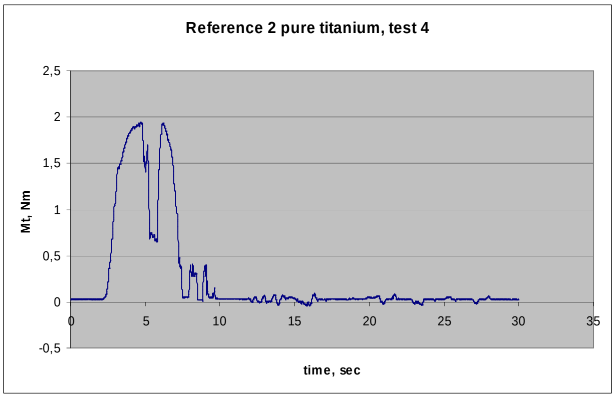

Fig. 12. Experimental torque, 2-nd not treated implant, test 4.

In case of the second reference dental implant made of pure titanium, the tests were done using an aluminium bar instead of the pig bone for blocking the implant and crack it. Furthermore, two adaptors made of C120 hardened steel for molds were manufactured.

In order to avoid the possibility to deform the hexagonal surfaces of the dental implant and the adaptor, another external cylinder to cover both cylindrical head of the dental implant and cylindrical head of the adaptor was manufactured.

Before making the measurements during the torsion test of the implant a test to verify the measurement range of the dynamometer was done (Fig. 9). The aim was to verify the possibility to measure a torsion moment for cracking the dental implants, estimated to be 6-10 Nm.

Consequently, the insertion of second reference dental implant made of pure titanium was made into a hole made in an aluminium bar, with the adaptor made of C120 hardened steel and external cylinder. It can be observed there were done 7 twists and torsion moment did not have values above 1.92 Nm. Hexagonal surface of the adaptor cracked partially, breaking in the next experiment, presented in Fig. 11.

Of course, the adaptor was changed and the tests continued.

The second adaptor made of C120 hardened steel resisted, but the aluminium bar ceased (Fig. 12). The implant was twisted by means of a gripper, the dental implant thread being destroyed, but even in these conditions the torsion moment did not exceed 3.1 Nm (Fig. 13).

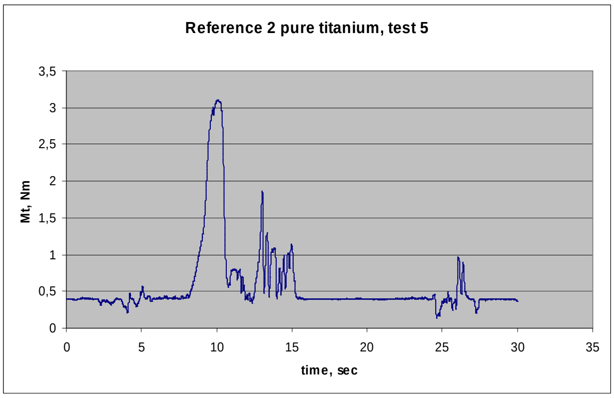

Fig. 13. Experimental torque, 2-nd not treated implant, test 5.

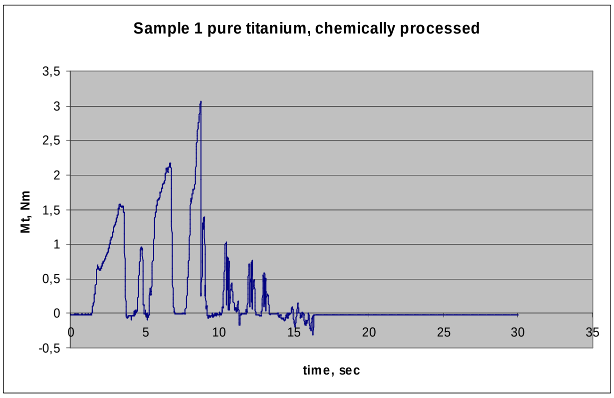

Fig. 14. Experimental torque, treated implant.

Only for information, a single experiment was done for the first dental implant made of pure titanium chemically processed, in the conditions of the previous experiment (Fig. 13), obtaining the graph in Fig. 14. The result was strictly similar. The torsion moment value never reached 6 Nm.

2.3. Conclusions of the tests

After all these experiments the following conclusions can be stated:

1. In practice there is no possibility to force dental implants in the presented experimental conditions.
2. Breaking of the pure titanium dental implant occurs at last, after the deterioration of the inserting tools and/or the bone in which the insertion is made.
3. Deterioration of the bone is out of question, because the insertion is done in a threaded hole.
4. Because the resistance of the alloyed titanium is double compared to that of the pure titanium, there is no problem of the resistance of the implants made of alloyed titanium.

At last, it is considered that the resistance of the titanium dental implant is so great that it is not influenced by diminish of the dimensions due to the chemical processing nor by the superficial layer resulted.

3. CRISTALLOGRAPHY TESTS OF TITANIUM IMPLANTS

There are different manufacturing methods of the titanium implants: cutting from rolled bars, forming, and rapid prototyping.

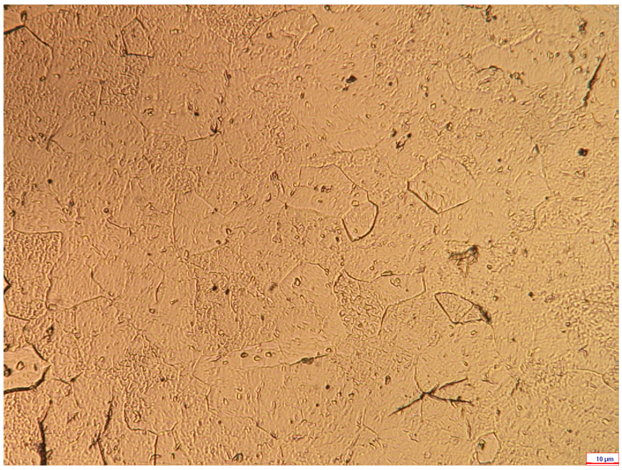

Fig. 15. Crystallographic structure of TiAl6V4 rolled bar (800×).

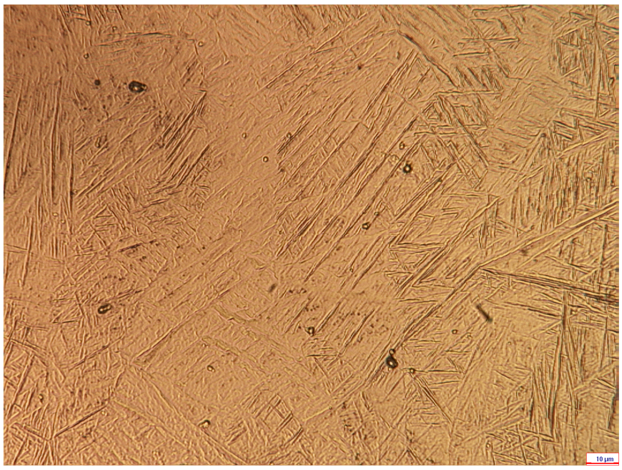

Fig. 16. Crystallographic structure of TiAl6V4 laser sintered rapid prototyping sample (800×).

The presented following tests were done in order to see if newly appeared rapid prototyping personalized titanium implants have more porous structure than those manufactured from rolled bars. This question was motivated by the very rapid prototyping technology, which deposits material point by point, every point being melted and welded to the rest of material.

The National Institute for Research and Development in Mechatronics and Measurement Technique has new equipment for rapid prototyping laser sintering of titanium powder. This is the reason it is interested in manufacturing personalized implants using this technology.

In order to compare the two materials proposed, rolled bar and rapid prototyping sample, the authors made the tests of crystallographic structure at University “Politehnica” of Bucharest, Faculty of Materials Engineering.

Figure 15 presents the microstructure of a rolled bar made of TiAl6V4. This material is used by Romanian manufacturer of dental implants and other implantable devices, Tehnomed Impex Co S.A.

In comparison, Fig. 16 presents the microstructure of a sample of TiAl6V4 obtained by laser sintered rapid prototyping. It can be seen the directions of laser depositions layer after layer.

Even if the initial aim of these tests was to see how porous the micro structure of the samples is, there are also other things to be seen.

It can be observed there are more pores in the rolled bar micro structure than in rapid prototyping structure. This could be explained by characteristics of the two technologies: rolling and rapid prototyping. Of course, rapid prototyping obtains finer structures than rolling because of point after point deposition, which leads to a more uniform structure.

It is very interesting to see the micro crystals in Fig. 15 and not seeing them in Fig. 16. Rapid prototyping technology involves layers deposition along different directions layer after layer, which leads to a reinforcement of micro structure.

It could be interesting to make tensile tests of the two types of material structures, to see the difference. This is a future research direction.

4. CONCLUSIONS

This paper presents engineering tests on titanium implants of two different types:

1. Dental implants made either of pure titanium, grade IV or alloyed titanium TiAl6V4, manufactured by Romanian producer of dental implants and other medical implantable devices, Tehnomed Impex Co S.A. Bucharest. These implants were manufactured by cutting operations and chemically processed in the end.
2. Orthopaedic implants made of alloyed titanium TiAl6V4, manufactured by the National Institute for Research and Development in Mechatronics and Measurement Technique of Bucharest. These implants were manufactured by laser sintered rapid prototyping.

All tests were done in POLITEHNICA University of Bucharest, in the laboratories of Machines and Manufacturing Systems Dept. and in the Faculty of Materials Science.

For tests regarding torsion moment of the dental implants made of pure and alloyed titanium, the following conclusions can be presented:

1. Tests were done in order to obtain the capable torsion moment of the implants, chemically processed or not. This meant the authors wanted to crack the implant and see the value of the torsion moment. The question to be answered was if the chemically processed implants have less resistance than the not processed ones. The possible reasons of a smaller resistance could be the thinner wall of the chemically processed implant and the chemically modified surface of the implant.
2. Because resistance of the alloyed titanium is twice the resistance of the pure titanium, it was not necessary to test implants made of TiAl6V4, because implants made of pure titanium had enough resistance;
3. The experimental conditions were much harder than in the real case, experimentally trying to screw the implants into unthreaded holes made into pig hard bone or aluminium. Even in these conditions the authors did not crack the pure titanium dental implant, both chemically processed or not. Furthermore, the inserting tools ceased, even if they were made of hardened steel for molds (usually, they are made of alloyed titanium).

Finally, the conclusion is that the resistance of the titanium dental implant, either pure or alloyed, is so great that it is not influenced by diminish of the dimensions due to the chemical processing nor by the superficial layer resulted.

Tests regarding the microstructure of the different possible methods to obtain the material implant were made with the aim to see how porous is the material, considering that pores are stress concentrators. The two considered methods to obtain the implant's material were rolling – in case the implant is manufactured by cutting – or laser sintering rapid prototyping.

Two important conclusions revealed:

1. There are more pores in rolled bar used as raw material for dental implants made by cutting than in the implants made by laser sintering rapid prototyping. This could be explained by the difference between the two technologies, rapid prototyping obtaining finer micro structure.
2. Micrograph of the implant made by laser sintering rapid prototyping revealed a reinforced micro structure of the material, with strings along different directions for each layer, as this method deposits point by point. This could mean the tensile resistance of the implant could be greater than the resistance of the rolled bars of titanium.

ACKNOWLEDGEMENT: Part of the work presented in this paper was supported by Romanian Ministry of Research and Education in the framework of the Program Research of Excellence, contract No. 71-057/18.09.2007.

REFERENCES

[1] S.M. Croitoru, D. Oprea, *Experimental Research on Measuring Cutting Efforts During Aluminium Alloys Drilling*, Proceedings of the 16-th International Conference on Manufacturing Systems ICMaS 2007, Published by Editura Academiei Române, ISSN 1842-3183, Bucharest, Romania, pp. 53-56.

[2] C. Minciu, S.M Croitoru, *Experimental research on cutting efforts at zinc alloys drilling and milling*, Academic Journal of Manufacturing Engineering , ISSN 1583-7904, 2009, Sibiu, Romania.

[3] A.I. Pacioga, *Cercetări privind dezvoltarea unei noi baze conceptuale în designul şi fabricarea protezelor de şold prin personalizarea lor* (Research on the development of a new conceptual base in the design and manufacture of hip prosthesis through customization), PhD Thesis, University "Politehnica" of Bucharest, 2011.

[4] * * * ASTM F67 *Standard Specification for Unalloyed Titanium, for Surgical Implant Applications*;

[5] * * * SR ISO 5832-2/1996 *Implanturi chirurgicale. Produse metalice. Partea 2:Titan nealiat* (Implants for surgery. Metallic materials. Part 2: Unalloyed titanium) IDT ISO 5832-3:1993.

[6] * * * SR ISO 5832-3/1996 *Implanturi chirurgicale. Produse metalice. Partea 3: Aliaj deformabil titan-aluminiu 6-vanadiu 4* (Implants for surgery. Metallic materials. Part 3: Wrought titanium 6-aluminium 4-vanadium alloy), IDT ISO 5832-3:1990.