International Journal of Innovative Studies in Sciences and Engineering Technology (IJISSET)

ISSN 2455-4863 (Online) **www.ijisset.org** *Volume: 5 Issue: 11 | 2019*

Mechanical Behaviour of the Ti10Ta Alloy Produced by Additive Manufacturing

Teresa Morgado[1, 2, 3], Catarina Valente[4], Josu Leunda[5], Alexandre Velhinho[6, 7]

1. LNEC – Laboratório Nacional de Engenharia Civil, Lisboa/ Portugal,

2. IPT – Polytechnic Institute of Tomar, Tomar/ Portugal,

3. UNIDEMI – Research and Development Unit for Mechanical and Industrial Engineering Universidade Nova de Lisboa, Portugal,

4. DEMI – Mechanical and Industrial Engineering Department, FCT NOVA – Faculty of Sciences and Technology, Universidade Nova de Lisboa, Portugal,

5. IK4-TEKNIKER - Advanced Manufacturing Technologies Unit, Polo Tecnológico de Eibar, Calle IñakiGoenaga 5, 20600 - Eibar (Gipuzkoa)/ Spain,

6. CENIMAT – Materials Research Center, Universidade Nova de Lisboa, Portugal,

7. DCM – MaterialsScienceDepartment, FCT NOVA – Facultyof Sciences andTechnology, Universidade Nova de Lisboa, Portugal,

Abstract: *The objective of this work concerns the study of the mechanical behaviour of a Titanium-Tantalum (Ti-Ta) alloy, Ti10Ta, produced by additive manufacturing. The study is motivated by the current lack of technical and scientific knowledge concerning the mechanical properties of this alloy, specifically LASER claddingTi10Ta.*

The present study describes the manufacturing process of titanium alloy specimens with a composition of 10 wt.% (weight percentage) of tantalum, fabricated as large blocks by LASER cladding and subsequently cut by wire-EDM(wire Electrical Discharge Machining), as well as reporting the experimental determination of the alloy mechanical properties.

Hardness, tensile and fatigue tests were performed. From these tests the Vickers hardness of the alloy was determined. Also, the tensile stress-strain curves were established, allowing the estimation of the alloy's Young´s modulus, yield and tensile strength together with the corresponding strains, strain after fracture and the cross-sectional area reduction, leading to conclude about the ductile behaviour of the alloy. Additionally, the equation for the uniform plastic flow regime of the material was established. As for the fatigue behaviour, the S-N curve was obtained, considering 3 levels of maximum applied stress, and the equation associated to this curve was established.

Keywords: *Ti10Ta Alloy, LASER cladding, Wire-EDM, Vickers Hardness, Mechanical Proprieties, S-N curves*

1. INTRODUCTION

Knowledge about the mechanical properties of the various types of Ti-Ta alloys is scarce ([1],[2],[3]), and does not cover the fatigue behaviour of the material. The documented information is insufficient, particularly for the specific case of Ti-Ta containing 10 % (wt% - weight percentage) tantalum. The scarcity of information is even more striking when the Ti10Ta alloy is produced by the emerging process of LASER cladding. One of the few studies approaching Ti-Ta alloys produced by LASER cladding, besides the one described in this paper, is the one by Morgado *et al.* [4], in which an experimental study is developed to evaluate the wear behaviour of the Ti-30%Ta and Ti-52%Ta (wt%) alloys. In what concerns the applicability of Ti-Ta alloys produced in various compositions by various methods, most of the studies found([5] and [6], for example)are about biomedical applications, dealing with corrosive resistance and biocompatibility behaviour of these alloys.

Concerning the production method used to fabricate the alloy studied in the present work, LASER cladding is a technique that, besides its additional application in the manufacturing of 3D components, finds an important use in the improvement of the mechanical properties of a material's surface through the addition of thin layers of different materials [4].LASER cladding uses a LASER beam as the heat source to melt and deposit a thin layer of a specified material with the desired properties onto a substrate [7],[8]. The area to be clad is heated by the absorption of energy delivered by the LASER beam. The heat input provided by a high-power LASER beam (most common power levels for industrial applications are above 5 kW) is spatially confined and very intense, thus increasing the heating rate of the surface layer. After the passing of the LASER, the heated surface layer becomes quenched, due to heat conduction to the cooler regions of the bulk. High rates of heating/cooling in the surface layer result in significant metallurgical modifications, such as grain refinement, formation of meta stable phases and/or micro structural transformation. The LASER cladding process traditionally uses CO_2 or various types of Nd: YAGLASER sources, although, more recently, the use of fibre LASER is becoming increasingly common[9],[10]. The cladding material can be transferred to the substrate by powder injection (the method used in this work to produce Ti10Ta alloy), wire feeding or paste feeding (all methods of continuous application of the cladding material during the process) and also by pre-depositing the loose powdered cladding material at the substrate's surface[7],[9].

The objective of the present work is to study the mechanical behaviour of the Ti10Ta alloy produced by the LASER cladding process, including its fatigue behaviour through the determination of the S-N curves. This study also includes the manufacturing process, research the selected process parameters to the Ti10Ta alloy manufacturing process.

To carry out that the stated purpose, the mechanical characterization of the alloy was performed, comprising a set of hardness, tensile and fatigue tests. The standard specimens of the Ti10Ta alloy used were produced by the LASER cladding technology with the continuous feeding/deposition of powders assisted by a LASER heat source. Specimens were extracted from the LASER cladding Ti10Ta blocks by wire-EDM (wire-Electrical Discharge Machining) cutting.

2. MATERIAL AND EXPERIMENTAL PROCEDURE

2.1 Material: Ti10Ta alloy

Titanium grade 2 plates of 15x160x160 mm were used as the substrate onto which the powdered cladding/coating material was deposited by LASER cladding. To form the Ti10Ta alloy that corresponded to the cladding material, titanium and tantalum powders were mixed in a weight percentage of 90% to 10%, respectively. These powders were supplied by *New Met Ltd. – New Metals and Chemicals Ltd* in a range of 45-90 µm and a purity of 99.9%. Both powders were produced by the so-called hydrogenation-dehydrogenation (HDH) process, presenting a crushed particle shape. The powders were premixed and kept in an oven at 100ºC for 20 hours before putting them in the powder hopper. The mixture was then kept for two hours in the hopper of the powder feeder, shaking it by an internal mixer. This procedure was necessary to guarantee the correct flow ability of the powder mixture, as the tantalum powders tend to agglomerate and produce clogging inside the conducting pipes.

2.2 LASER Cladding Process

A 2.2 kW diode pumped Nd: YAGLASER was used for the cladding process. The LASER beam was guided to the working region by means of an optic fibre and through a coaxial cladding head. This head allows varying the LASER spot on the work zone by changing the position of the focusing lens. The spot size was set to be of 2.7 mm on the working plane. The LASER head was fixed into a six-axis robot arm, which produced the relative motion between the LASER head and the substrate. The powder mixture was carried to the LASER head by means of a powder feeder, using argon as a carrier gas.

To adjust the process parameters for producing defect-free coatings, an initial experimental batch was carried out, where the LASER power, the powder feed-rate and the shielding gas flow rate were varied. 4-layer coatings were deposited with each set of parameters, overlapping 20 tracks for producing each layer, with a zigzagging motion, where the direction was rotated by 90º from layer to layer.

To analyse the coatings, cross sections were extracted by precision abrasive cutting to ensure that all sections were perpendicular to the plane on which the cladding layers were built. The freshly cut surfaces were processed by SiC-based grinding and diamond suspension-based polishing down to $R_a < 0.01$ µm. The metallurgical characterization of the samples was based on optical microscopy inspection, to study the inner quality of the coatings. No cracks were observed in any of the coatings, and the adherence to the substrate was also solid, with enough dilution. On the other hand, porosity issues were detected in most of the coatings. Even though such issues could not be completely eliminated, the parameters of the coating with minimal porosity, shown in Table 1, were selected for producing the final blocks.

Table 1- *Process parameters used for producing the final coatings by LASER cladding*

LASER Cladding Process Parameters	
LASER power (kW)	0.7
Scanning speed (mm/s)	15
Spot diameter (mm)	2.7
Powder feed rate (g/min)	3.95
Overlap distance (mm)	1.0
Height offset (mm)	0.2
Shielding gas	He
Shielding gas flow (l/min)	20

Two blocks of Ti10Ta where produced, with dimensions of roughly 120x120x10mm, by depositing 50-layer coatings with 120 overlapped tracks in each layer. From these blocks, 18 specimens and 3 samples of Ti10Ta were extracted to develop the experimental study of this work.

2.3 Wire - EDM Cut

From the 2 Ti10Ta blocks produced by LASER cladding, the 18 tensile specimens were extracted with their geometry normalized in accordance with *ASTM E8/E8M-09*[11]. The cutting pattern used on the blocks, together with one of the extracted specimens ,is shown in Figure 1. Cutting was performed by wire-EDM, with a brass wire, using an *ONA PRIMA E-400 + AWF* machine.

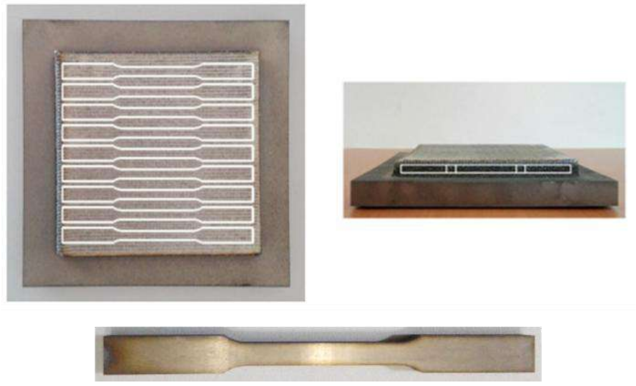

Figure 1*-Wire-EDM cutting pattern in one of theTi10Ta blocks produced by LASER cladding. Acut specimen is also shown.*

The relevant parameters used for the wire-EDM cutting operation are summarized in Table 2:

Table 2- *Cutting parameters used for wire-EDM operation*

Wire-EDM Cutting Parameters	
Applied current (A)	10
Applied voltage (V)	110
Wire speed (mm/s)	7
Tensile force on the wire (N)	18

2.4 Mechanical Characterization Tests

After a grinding and polishing treatment as prescribed in the *ASTM E3-95* standard [13], Vickers micro hardness tests were performed on 3 samples of Ti10Ta, in accordance with *ASTM E384-16* [12]. These tests were executed in *Mitutoyo HM-112*Micro hardness 6 indentations being made on each sample.

Uniaxial tensile tests were performed over 6 specimens at room temperature, with a stroke rate of 3 mm/min, as prescribed by *ASTM E8/E8M-09* [11],using a *Shimadzu AG-50kNG* universal testing machine. The tests were also conducted at room temperature.

Additionally, fatigue tests were performed on the 12 remaining specimens, using an *Instron 1342* universal testing machine. The tests were also conducted at room temperature [11]. One of 3 levels of maximum stress (650 MPa, 550 MPa and 450 MPa)was used for each group of 4 specimens. The stress cycle applied was sinusoidal, a stress ratio of 0.5 being selected.

3. RESULTS AND DISCUSSION

The value estimated for the Vickers hardness of the Ti10Ta alloy produced by LASER cladding, after the calculation of the average from the 3 samples, is $255.2^{+4.4}_{-3.7}$ HV0.5. However, it should be noted that, due to the porosity found in the samples, this result may be a lower estimation for the intrinsic hardness value of the sound material.

The measured tensile properties of the alloy Ti10Ta are summarized in Table3, which shows the average Young's modulus, yield strength and tensile strength, along with the corresponding yield and uniform tensile strains. The small value measured for the Young's modulus (9.77 GPa in terms of true value) for the Ti10Ta alloy is similar to that exhibited by the human bone (5 to 30 GPa) [14]. This result may be a consequence of the presence of tantalum in the alloy, given that this element, as identified by Song *et al.* [2], when alloyed within the Ti-Ta system, has the potential to promote a decrease of the Young's modulus, while also increasing the tensile strength in comparison with the non-alloyed titanium. It was also referred by Kunčická *et al.* [14] that to reduce the Young´s modulus of Ti6Al4V, mainly used as biomaterial, tantalum is one of the elements able to replace vanadium, an alloying element responsible for increased stiffness. This effect can eventually be verified in the reduction of the Young's modulus of a Ti-Ta alloy. Apart from the chemical composition, Kunčická *et al.* also mentioned other factors contributing for a lowered stiffness, such as the presence of porosities, which in the present case could also contribute to the low Young's modulus obtained for this Ti10Ta alloy.

Table3 *Average values of Ti10Ta alloy tensile properties.*

	Engineering values	**True values**
Young's Modulus [GPa]	8.35	9.77
Yield Strength [MPa]	642	692
Yield Strain	0.0771	0.0743
Tensile Strength [MPa]	735	855
Uniform Tensile Strain	0.164	0.152

In an effort to ascertain the LASER clad Ti10Ta alloy ductility, the strain after fracture and the cross-sectional area reduction of the tested specimens were measured in the present work, the resulting values are presented in Table4.

Table 4- *Values of the strain after fracture and of the cross-sectional area reduction of the tested specimens.*

	Strain after Fracture	**Cross Sectional Area Reduction**
Average	0.168	0.228
Average (%)	16.8	22.8

To Callister and Rethwisch [15], fragile materials have a strain after fracture smaller than c. 5%. Given the measured values, the conclusion reached is that the tested Ti10Ta alloy exhibits a ductile behaviour. Furthermore, the average value obtained for the cross-sectional area reduction reinforces this conclusion, given that according to Barralis e Maeder [16] fragile behaviour is common for section reductions smaller than 10%. Also, the tensile curves presented in Figure 3 already indicated a ductile behaviour for the material, since they demonstrated the existence of significant plastic deformation before the occurrence of fracture, as is characteristic of ductile materials [17].

The results of the Ti10Ta tensile tests also provided data to represent in a bilogarithmic graphic the equation (1),that translates the true stress - true strain ($\bar{\sigma}$-$\bar{\varepsilon}$) relationship of the material in the uniform plastic deformation regime. Such representation, shown in Figure 3, corresponds to a line of slope n, according to equation (2)

$$\bar{\sigma} = K\bar{\varepsilon}^{n}, \tag{1}$$

$$\ln(\bar{\sigma}) = \ln(K\bar{\varepsilon}^{n}) \Rightarrow \ln(\bar{\sigma}) = \ln(K) + n\ln(\bar{\varepsilon}). \tag{2}$$

Thus, it becomes possible to estimate the values of the hardening (n) and resistance (K) coefficients, which define the material's plastic flow behaviour under constant temperature and strain rate conditions.

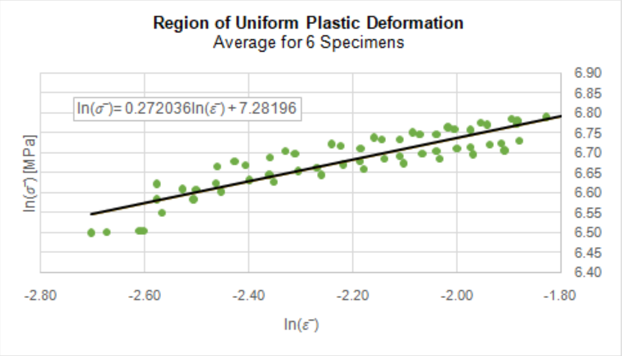

Figure 3- *Bilogarithmic representation of the uniform plastic deformation regime, as recorded by the true stress- true strain* ($\bar{\sigma}$-$\bar{\varepsilon}$) *curve, for determination of* n *and* K.

As mentioned, the analysis of the data led to estimate the following values:$n \cong 0.272$; $\ln(K) \cong 7.28196 \Rightarrow K \cong e^{7.28196} \Rightarrow K \cong 1454$ MPa.

Therefore, equation (3) can be used to describe the LASER clad alloy's behaviour under a uniform plastic deformation regime:

$$\bar{\sigma} = 14534\bar{\varepsilon}^{0.272} \tag{3}$$

The S-N curve was determined from the tests performed, considering the points corresponding to the calculated average number of cycles (N) during which the material resisted to fracture when subjected to each level of maximum applied stress, σ_{max} , (650 MPa, 550 MPa or 450 MPa).The fatigue behaviour of the Ti10Ta alloy may be characterized by the logarithmic equation (4):

$$log\sigma_{max} = -0.181\, logN + 3.53. \tag{4}$$

Hence, the relationship between the applied stress and the number of cycles during which fatigue fracture may be hoped to be averted may be translated by equation (5)

$$\sigma_{max} N^{0.181} = 3369. \tag{5}$$

4. CONCLUSIONS

A new alloy of Titanium and Tantalum was manufacture by LASER Cladding technology. The manufactured parameters were obtained. As Titanium and Tantalum have very different fusion points these parameters are very difficult to obtain and important to guarantee a homogeneous alloy.

This paper responds to the reduced technical-scientific knowledge identified in what concerns the mechanical behaviour of Ti-Ta alloys, particularly in the case of Ti10Ta produced by LASER cladding. In addition, it was found that porosity constitutes one of the defects resulting from producing the alloy under the selected set of process parameters and with the type of powders used.

REFERENCES

[1] Y. L. Zhou, M. Niinomi, and T. Akahori, "Effects of Ta content on Young's modulus and tensile properties of binary Ti-Ta alloys for biomedical applications", *Mater. Sci. Eng. A*, vol. 371, n. 1–2, pp. 283–290, 2004.

https://doi.org/10.1016/j.msea.2003.12.011

[2] Y. Song, D. S. Xu, R. Yang, D. Li, W. T. Wu, and Z. X. Guo, "Theoretical study of the effects of alloying elements on the strength and modulus of β-type bio-titanium alloys", *Mater. Sci. Eng. A*, vol. 260, n. 1–2, pp. 269–274, 1999.

https://doi.org/10.1016/S0921-5093(98)00886-7

[3] Y. L. Zhou, M. Niinomi, T. Akahori, M. Nakai, and H. Fukui, "Comparison of Various Properties between Titanium-Tantalum Alloy and Pure Titanium for Biomedical Applications", *Mater. Trans.*, vol. 48, n. 3, pp. 380–384, 2007.

https://doi.org/10.2320/matertrans.48.380

[4] T. L. M. Morgado, H. Navas, and R. Brites, "Wear study of Innovative Ti-Ta alloys", *Procedia Struct. Integr.*, vol. 2, pp. 1266–1276, 2016.

https://doi.org/10.1016/j.prostr.2016.06.162

[5] J. Kesteven, M. B. Kannan, R. Walter, H. Khakbaz, and H. C. Choe, "Low elastic modulus Ti-Ta alloys for load-bearing permanent implants: Enhancing the biodegradation resistance by electrochemical surface engineering", *Mater. Sci. Eng. C*, vol. 46, pp. 226–231, 2015.

https://doi.org/10.1016/j.msec.2014.10.038

[6] Y. Liu *et al.*, "Synthesis of Ti-Ta alloys with dual structure by incomplete diffusion between elemental powders", *J. Mech. Behav. Biomed. Mater.*, vol. 51, pp. 302–312, 2015.

https://doi.org/10.1016/j.jmbbm.2015.07.004

[7] E. Toyserkani, A. Khajepour, and S. F. Corbin, *LASER Cladding*. 1st Edition, CRC Press,USA, 2004.ISBN 9780849321726

[8] J. M. S. P. Torres, “Improvement and Automatization of a LASER Cladding System” in Portuguese, Master Thesis, FCT, UNL – Universidade NOVA de Lisboa, Portugal, 2015.

[9] M. Schneider, “LASER CLADDING WITH POWDER: effect of some machinig parameters on clad properties”, Ph.D. Dissertation, Universite it Twente, The Netherland, 1998.

[10] V. Malin and S. Woods, “Efficient high-power diode LASER cladding - Industrial LASER Solutions”. [Online]. Available: http://www.industrial-lasers.com/articles/print/volume-24/issue-8/features/application-report/efficient-high-power-diode-laser-cladding.html. [Accessed: 02-Jan-2017].

[11] ASTM E8/E8M-09, “Standard Test Methods for Tension Testing of Metallic Materials”, ASTM International, USA, 2009.

[12] ASTM E384-16, “Standard Test Method for Micro indentation Hardness of Materials”, ASTM International, USA, 2016.

[13] ASTM E3-95, “Standard Practice for Preparation of Metallographic Specimens”, ASTM International, USA, 2001.

[14] L. Kunčická, R. Kocich, and T. C. Lowe, “Advances in metals and alloys for joint replacement”, *Prog. Mater. Sci.*, vol. 88, pp. 232–280, 2017.

[15] W. D. Callister, Jr. and D. G. Rethwisch, *Science and materials engineering: an introduction* in Portuguese, 9th ed. Brasil: LTC, 2016.

[16] J. Barralisand G. Maeder, *Metallurgical Promptuary: elaboration, structures-properties and standardization* in Portuguese, 3rd ed. Portugal: Fundação Calouste Gulbenkian, 2014.

[17] C. A. G. de M. Branco, *Mechanics of Materials* in Portuguese, 5th ed. Portugal: Fundação Calouste Gulbenkian, 2011.