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# Correlative microscopy analysis of surface topography in machining Ti-6Al-7Nb

Ana Horovistiz\*, Sílvia Carvalho, António J. Festas, J. Paulo Davim

TEMA, Department of Mechanical Engineering, University of Aveiro, 3810-193, Aveiro, Portugal

\* Corresponding author. Tel.: +351 234 370 830; fax: +351 234 370 985. *E-mail address:* horovistiz@ua.pt

## Abstract

Titanium alloys, namely Ti-6Al-7Nb, are used in the biomedical industry. The study of surface topography is crucial for the development of medical components. The objective of this work is to propose the correlative microscopy technique developed for the analysis of surfaces machined by the turning process of Ti-6Al-7Nb alloy. This technique was based on the association of the 3D reconstruction by extended depth-of-field method from Optical Microscopy (OM) with the Scanning Electron Microscopy (SEM) and microanalysis modes. The correlative microscopy allows a correspondence between the cutting conditions and the material properties, through the analysis of the machined surface.

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## 1. Introduction

Machining of biomedical components is of major importance for modern industry since the improvement of the life quality of the world population has required the development of engineering products and techniques for medical purposes [1], such as prostheses and implants of high durability, without aesthetic impacts and with low patient rejection.

Among the various materials currently used for the manufacturing of biomedical components are Ti alloys. These materials stand out for its excellent mechanical and corrosion properties and are much less dense than stainless steel. Nowadays, the most commonly used titanium alloy is Ti-6Al-4V. However, the possible toxicity [2] of this alloy, due to the presence of vanadium (V), prompted the development of substitute alloys, such as Ti-13Nb-13Zr, Ti-5Al-2.5Fe, Ti-6Al-7Nb, etc.

Some research work has already been carried out about

machinability, tribological properties and microstructural evolution of Ti-6Al-7Nb alloy [3-5]. However, more studies are still needed for the understanding of manufacturing of biomedical components techniques, namely the machining process. Surface topography is a feature that exhibits great potential for understanding and optimizing machining processes. In this direction, modern microscopy techniques are useful tools in inspecting roughness, since they can quantitatively relate the topographic patterns of the machined surfaces to the cutting parameters and the material properties.

The correlative microscopy, a methodology that consists on evaluating the same field by different microscopy techniques, has been gaining space in the scientific field of material characterization combining the advantages of different microscopy techniques and chemical analysis [6].

Among the possible combinations, the optical microscopy (OM) together with scanning electron microscopy (SEM), has been used for a complete inspection of microstructures in terms of a large range of surface properties. The SEM is associated with high spatial resolution and the possibility of allying semi-

quantitative chemical analysis (EDS) techniques. However, it presents a restricted field of vision, high operating cost and expensive sample preparation. The OM has the advantage of having a larger field of view that facilitates identification and location of objects of interest (phases, precipitates, pores, cracks, etc.), low preparation and operation cost. It is, however, limited by lateral resolution at high magnifications. The blend of these two techniques of microscopy may result in the combination of the best of both.

The correlative microscopy has been used for the analysis of fracture surfaces of different materials, providing general information on the mechanisms involved in fracture processes [7], with the possibility of associating different resolution scales and contrast modes. Inspection of machined surface topography may also benefit from correlative microscopy since this technique provides a wide range of information on topography patterns generated by the different machining parameters, eventually associated with microstructural modifications. These relationships are most obvious on dual-phase ( $\alpha + \beta$ ) titanium alloys due to the widely recognized effect of low thermal conductivity on phase transformations during machining processes [8].

The major challenge for the success of correlative microscopy lies on the perfect matching of observed regions in the different microscopes [9]. This matching requires the establishment of a common spatial reference system for the involved microscopes, combined with software tools based on digital image processing.

This work proposes the correlative microscopy technique developed for the analysis of machined surfaces by the *V-Free* Ti-6Al-7Nb alloy turning process, based on the association of the 3D reconstruction by extended depth-of-field method from OM with the imaging modes and microanalysis from SEM. The solution consists of a two-dimensional scaled sample holder, developed for this study, associated with Vickers indentations on the sample surface, to establish the common spatial reference and plugins for NIH ImageJ [10], a public domain Java image processing and analysis program. The software allows the fusing of elevation maps based on OM image stacks to the SEM images for the same region of the machined surface. Thus, this work also intends to contribute to expanding the set of useful tools for quantitative and qualitative inspection of machined surface topography. In this manner, the correlative microscopy is presented here as a tool for evaluation of machined surfaces.

## 2. Experimental procedure

### 2.1 Machining experiments

A cylindrical bar of Titanium alloy, Ti-6Al-7Nb, with a diameter of 12 mm from TiFast S.r.l. was employed in the machining experiment. The material presented a chemical composition of aluminium (5.94–6.02%), carbon (0.013%), iron (0.15–0.16%), hydrogen (0.003%), nitrogen (0.005%), niobium (6.83–6.90%), oxygen (0.169–0.178%), tantalum (0.05%) and titanium (in balance). Ti-6Al-7Nb was one of  $\alpha + \beta$  duplex phases titanium alloy, no coarse, elongated  $\alpha$  platelets,

inclusion free. The alloy was extruded and then been subjected to heat treatment for stress relief annealing.

The turning tests were performed on a CNC turning center Kingsbury MHP 50 in dry condition. The (Ti, Al) N + TiN coated tools were employed with the tool holder (C3-PCLNL-22040-12). The parameters chosen to demonstrate the application of the correlative microscopy technique to machined surfaces are listed in Table 1, where:  $v_c$  is the cutting speed;  $f$  is the feed rate and  $a_p$  is the depth of cut. The  $R_a$ , the average of a set of individual measurements of surfaces peaks and valleys, is shown in this same Table. After the turning tests, one sample at each condition was cross-sectioned for topographic surface analysis. The length of the pieces was approximately 5 mm.

Table 1 Parameters used in the studies.

Cutting conditions	$v_c$ (m/min)	$f$ (mm/rev)	$a_p$ (mm)
as-received			
sample1	30	0.15	0.5
sample2	90	0.05	

### 2.2 Surface topography analysis

Each cylindrical sample was fixed in a bi-dimensional scaled sample holder, developed for this experiment. The sample was then marked on the machined surface with the aid of a Vickers diamond pyramid penetrator with a load of 200 gf for 10 seconds. The system, cylindrical specimen and sample holder was taken to an optical microscope, a Nikon-Eclipse LV150 reflected Optical microscope, with objective lenses for extended working distances. The indentation prism was used as a reference system in the picture and located in the microscope through the markings of the sample holder. The spatial resolution of the picture was fixed on 4000x3000 pixels. From this step, the selection of the parameters of microscopy and image acquisition had the criterion of optimizing the parameters for the extended depth-of-field method. A 20X objective was used to obtain 200X total magnification under brightfield illumination. These procedures are explained in detail in Horovistiz *et al.* [11].

The sample holder was then mounted on the scanning electron microscope, a Hitachi TM4000Plus, equipped with one Bruker Quantax 75 EDS. The SEM stage was then calibrated by finding the reference marks (Vickers impression), setting the microscope for 1000X magnification and using SE electron detector at 15 mm of working distance. Vickers impressions have regular shapes being very useful to check the consistency of alignment quality, through rotation and translation operations of the sample holder, having as reference the edges of the indentation. The Fig.1 show an example of operation. Finally, the image files corresponding to the pictures for each bright-field stack were loaded under the NIH Image J, driving the SEM stage to almost the same x, y fields pictured with the light microscope. The algorithm used in this case was “Stack Focuser” plugin, a solution proposed by Michael Umorin and distributed from NIH ImageJ website (<http://rsb.info.nih.gov/ij/plugins/stack-focuser.html>).

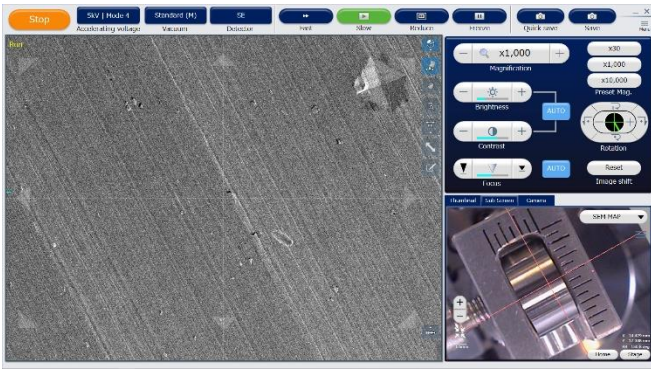


Fig.1 Illustration of SEM placement. In the lower right corner is the bi-dimensional sample holder.

Roughness measurement was performed using a Hommel TM Tester T1000 profilometer portable rugosimeter. The roughness of a cylindrical sample as-received was also measured before the turning operation for comparison.

### 3. Results and discussion

The main advantage of this technique is to combine the topographic information of the machined surfaces of the OM height maps to the distribution maps of chemical elements obtained by microanalysis in SEM images. Likewise, the aspects of topography or possible inclusions can be compared between SEM and reconstructed OM images.

The sample2 of Table 1 was used as an illustrative example of the fields matching in correlative microscopy. The results are presented in Fig.2: a) bright-field OM elevation map; b) image focused by extended depth-of-field method and; c) SEM image of the same field. Fig. 1d illustrates the 3D elevation map and it is possible to visualize the surface topography resulting of feed marks from the turning process at corresponding cutting conditions. Fig.2e permits the analysis of the correlation between the topography formation and any cracks formed during the turning process. It is known that microstructural modifications may occur during machining processes. This phenomenon can influence the topography of the surface. This way, Fig.2f offers a correlation between the OM 3D elevation map and the EDS map of chemical element distribution.

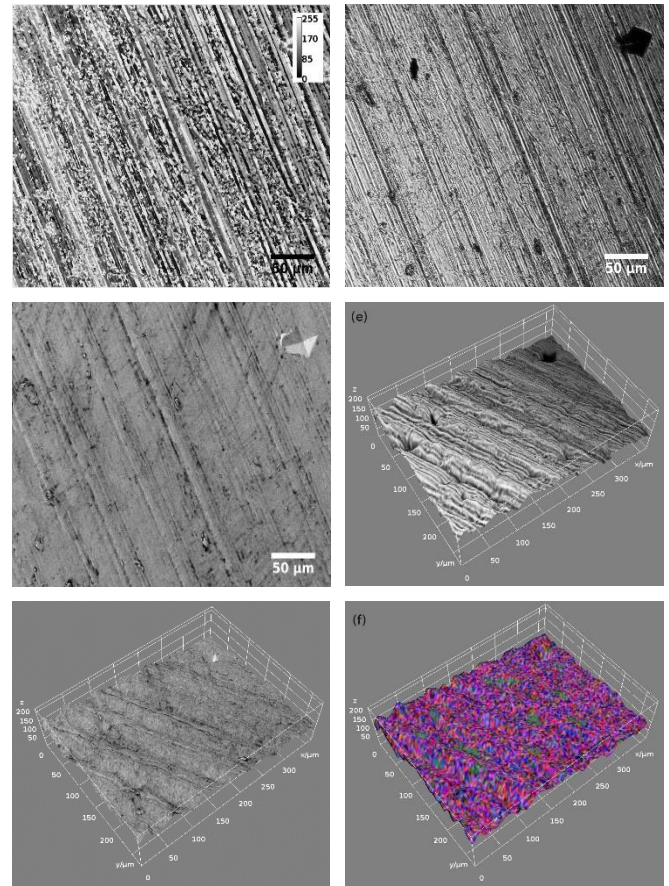


Fig.2 Example of the fields matching in correlative microscopy for sample 2 ( $vc= 90(m/min)$ ;  $f=0.05 (mm/rev)$  and  $ap=0.5 (mm)$ ) : (a) bright-field OM elevation map; (b) image focused by extended depth-of-field method ; (c) SEM image at 5eV; (d) 3D representation of height map; (e) 3D representation of height map with the SEM image as loaded texture; (f) 3D representation of height map with the corresponding EDS map as loaded texture: red, blue, green, color are representing, respectively, the distribution of Ti, Nb, and Al.

Fig. 3 illustrates the examples of 3D elevation maps and their corresponding EDS maps after turning tests under different cutting conditions (Table 1) where it is possible to visualize the topography surface with the resulting feed marks. The topography surface of a received sample is displayed for comparison. The roughness values,  $Ra$ , are shown in each illustration of 3D elevation map of Fig.3.

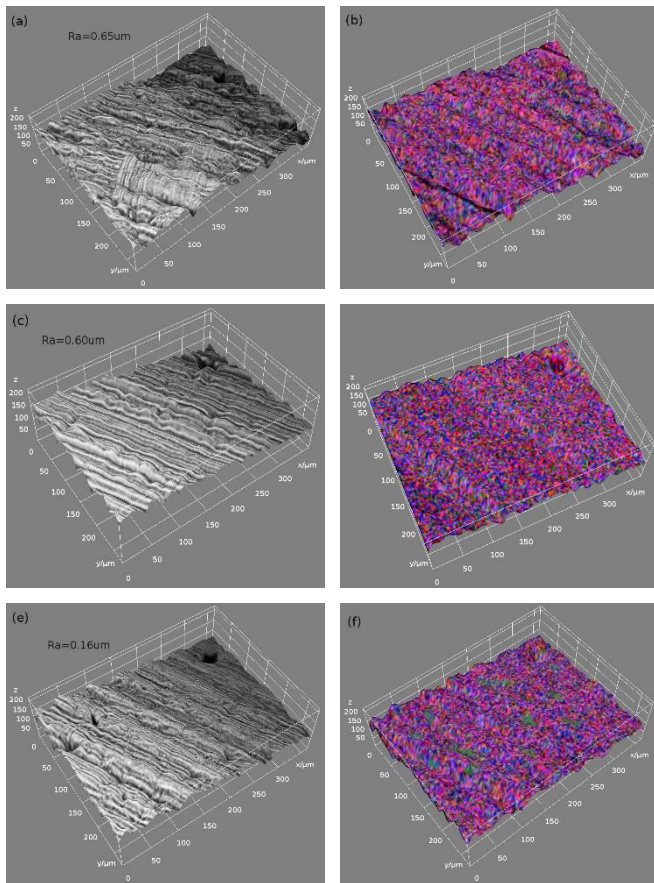


Fig.3 3D elevation map of turned surface of Ti-6Al-7Nb alloy samples (a, c and d) and its corresponding EDS map under different (b, d and e): a and b correspond to as-received sample; c and d correspond to sample 1 on Table 1 ( $v_c=30$ (m/min);  $f=0.15$  (mm/rev) and  $ap=0.5$  (mm)) and e and f correspond to sample 2 on Table1 ( $v_c=90$ (m/min);  $f=0.05$  (mm/rev) and  $ap=0.5$  (mm)).

The results presented in Fig. 2 correspond to sampling collected in random fields in each sample in order to compensate for possible heterogeneities. In fact, the heterogeneity may have consequences in the development of topography of the surface during the turning operation. The overall comparison of the results suggests that the cutting conditions affect the surface topography of the Ti-6Al-7Nb alloy samples. In Fig. 2 (c and e) it can be noted an evolution of the feed marks according to the variation of the feed rate,  $f$ , showing on Table1. These more regular topographies contrast with the more random surface topography patterns than the sample as-received displays (Fig 2a).

There seems to be a qualitative agreement between the roughness values measured,  $R_a$  and the topography displayed on the 3D representation of the height map (Fig.2 a, c and d). In addition, by observing the evolution of EDS maps (Fig.2 b, d and f) one can observe a change in the homogeneity of the distribution of the spots related to the chemical elements. In Fig.2f the green color, which corresponds to the element Al appears concentrated on small islands, apparently close to the depressions developed during the turning operation. Some works have reported modifications of phases and grain sizes on machined surfaces of Ti-6Al-4V titanium alloy [12-13]. The authors demonstrated that the cutting parameters such as cutting speed and depth of cut played a fundamental role in the

microstructural transformation.

It is evident that the understanding of this phenomenon will have to contemplate a global analysis of the turning process, such as temperature measurements, forces, and a detailed microstructural study of the Ti-6Al-7Nb alloy. However, the use of correlative microscopy, assisted by digital image processing, is an advantageous and promising technique for a semi-quantitative inspection of the phenomena that occurred during the machining processes.

#### 4. Conclusion

The technique of correlative microscopy associates directly aspects regarding the cutting conditions in the machining operations with the properties of the material, through the analysis of the machined surface, combining the advantages of SEM and OM. The results of the samples analyzed in this study allow us to infer that the cutting parameters influence the topography and the microstructure of Ti-6Al-7Nb since it shows modifications in the distribution of the chemical elements present in the alloy in preferential sites, related to the machining topographies patterns. Many surface and material properties can be evaluated similarly, regarding the range of information provided by the techniques associated with both OM and SEM. It is certainly necessary to carry out more experiments, analyzing a larger number of machined surfaces samples, different cutting conditions to rely on other quantitative analyzes in order to improve the reliability of the results. However, it is believed that new results will provide convincing evidence of the usefulness of correlative microscopy approaches of machined Ti alloy surfaces. It is also believed that such approach will continue to be increasingly used.

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