

# MULTIREFLEXION X RAY DIFFRACTION METHOD FOR RESIDUAL STRESS INVESTIGATION IN THE Ti-BASED BIOMATERIALS

MARIANNA MARCISZKO<sup>1\*</sup>, ANDRZEJ BACZMAŃSKI<sup>2</sup>,  
SEBASTIAN WRONSKI<sup>2</sup>, MIROSLAW WROBEL<sup>3</sup>,  
CHEDLY BRAHAM<sup>4</sup>

<sup>1</sup> ACMIN, AGH UNIVERSITY OF SCIENCE AND TECHNOLOGY,  
POLAND

<sup>2</sup> WFIS, AGH UNIVERSITY OF SCIENCE AND TECHNOLOGY,  
POLAND

<sup>3</sup> WIMIP, AGH UNIVERSITY OF SCIENCE AND TECHNOLOGY,  
POLAND

<sup>4</sup> PIMM, ENSAM ARTS ET MÉTIERS PARISTECH, FRANCE

\*E-MAIL: MARCISZKO@AGH.EDU.PL

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## Introduction

Titanium and its alloys are commonly used metallic materials for implants [1,2]. To improve mechanical performance of prosthesis different surface treatment can be applied [3]. The appropriate generated stress ( $\sigma$ ) can reduce crack nucleation and propagation and consequently improves fatigue resistance of the implant. Diffraction X-ray methods are commonly used for residual stress ( $\sigma$ ) determination [4]. However, standard methods are not advised for the analysis of heterogeneous stress state because the penetration depth ( $\tau$ ) of X-ray radiation varies significantly during measurement. Using proposed in this study MGIXD (multireflexion grazing X-ray diffraction) method it is possible to perform a non-destructive analysis of the heterogeneous stresses for different volumes below the surface of the sample.

## Materials and Methods

MGIXD [5,6] was applied for  $\sigma$  determination using laboratory X-ray diffractometer as well as synchrotron radiation for Ti and Ti-alloy (TABLE 1) subjected to different mechanical surface treatments (polishing and grinding).

TABLE 1. Composition of the materials used in presented study (wt. %).

Ti grade 2	Ti: bal. C: 0.010	O: 0.131 N: 0.01	Fe: 0.109 Ni: 0.02
Ti6Al4V	Ti: bal. C: 0.008	O: 0.20 N: 0.05	Fe: 0.25 Al: 6.0 V: 4.0

The laboratory X-ray diffractometer (X'Pert PANalytical) with Cu K $\alpha$  radiation, was equipped with Göbel mirror to collimate incident beam and the parallel plate collimator in the reflected beam optics. The synchrotron experiment was performed at HASYLAB, DORIS III storage ring, on beamline G3, using soller collimator. The double-crystal germanium monochromator was used. Using synchrotron radiation the  $\tau$  can be changed for the same incident angle ( $\alpha$ ) by changing wavelength ( $\lambda$ ). Three different  $\lambda$  (1.2527 Å, 1.5419 Å,  $\lambda = 1.7512$  Å) were chosen for this study and the  $\alpha$ , for which the penetration depth is the same, were calculated.

## Results and Discussion

First the depth-dependent  $\sigma(\tau)$  profiles were determined from measurements performed on laboratory diffractometer for different  $\alpha$ . The obtained results indicate that: stresses close to zero were measured for reference Ti powder, furthermore tensile  $\sigma$  were generated after grinding and compressive  $\sigma$  after polishing. No significant

evolution of stresses occurs in the depth-dependent stress profile in the case of Ti sample, while the significant gradient of stresses occurs for polished Ti alloy. The results obtained for Ti6Al4V were verified using synchrotron radiation. The example of obtained peak profile is presented in FIG. 1. Strong asymmetry suggests that in fact there are two irradiated regions in the sample with different microstructure. That is why the diffraction peaks were separated into two having different integral widths and position. The broad peak represents 'hard' deformed material in the layer and the narrow one the 'soft' base material.

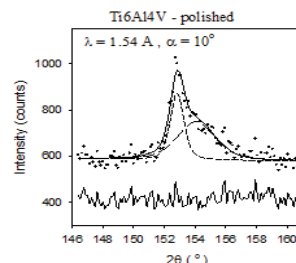


FIG. 1. The example peak profiles for the penetration depth  $\tau = 1.5$   $\mu$ m.

The obtained depth-dependent profile of stresses is presented in FIG. 2.

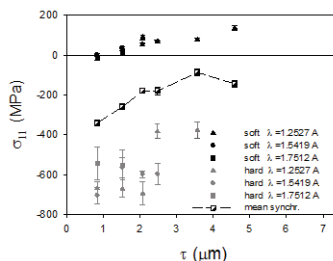


FIG. 2. The depth-dependent profiles of the  $\sigma$  for Ti6Al4V sample. Results after peak separation are plotted as the function of  $\tau$ .

High compressive stress of about 500-700 MPa has been found in the layer, while in the base material a small tensile stress increases with penetration depth within the range of about 0-120 MPa. A very good agreement of the results obtained using three different wavelengths of synchrotron radiations as well as the classical X-rays (Cu K $\alpha$  radiation) was found.

## Conclusions

The MGIXD is a non-destructive tool which can be successfully used for determination of depth-dependent stress distribution in Ti-based biomaterials. Such measurement is possible due to constant penetration depth of X-ray radiation in the studied material. Furthermore the information depth can be easily changed by setting different  $\alpha$ . As it was presented in the study the MGIXD method has very important advantages in comparison with other diffraction methods of stress determination especially curtail in the context of biomaterials.

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## References

- [1] S.R.Paital, N.B.Dahotre, Materials Science and Engineering R 66 (2009) 1–70.
- [2] A. Mitchell, P. Shrotriya, Wear 263 (2007) 1117–1123.
- [3] P.Peyre, A.Sollier, L.Berthe, E.Bartnicki, R.Fabbro, I.Chaieb and C.Braham, Eur. Phys. J. Appl Phys, 23 (2003) 83-88.
- [4] I.C.Noyan, J.B.Cohen, Residual Stress-Measurement by Diffraction and Interpretation, Springer-Verlag, Berlin, 1987.
- [5] S.J.Skrzypek, A.Baczmański, W.Ratuszek, E.Kusior, J. Appl. Cryst.34 (2001) 427- 435.
- [6] M.Marciszko, A.Baczmański, C.Braham, M.Wróbel, W.Seiler, S.Wróński, K.Berent, J. Appl. Cryst. 49 (2016) 85-102.