

Residual Stress on Titanium Substrate Prepared By Anodization Technique

Sami A. Ajeel

Metallurgy and Production Department, University of Technology
Al-Sinaa'a St, 52, Baghdad, IRAQ
Tel: +964- 7713014752 E-mail: samiabualnon2@yahoo.com

ABSTRACT

Residual stresses of pure titanium and anodizing titanium interlayer were measured using x-ray diffraction (XRD) method. The X-ray diffraction technique was used to provide residual stress measurements of pure titanium and anodized titanium.

XRD analysis also showed the presence of tensile stress in the titanium substrate while presence of compressive stress in the TiO₂nanotubes interlayer . The measured residual stresses in the TiO₂interlayer are -6.15 MPa and -8.8 MPa for anodized 10 V and 30 V respectively.

Keywords : tensile stress , compressive stress , nanotubes.

1. INTRODUCTION

Residual stresses can be defined as the stresses which remain in a material in the absence of any external forces. There are many stress determination methods. Some of those methods are destructive and some are nondestructive. X-ray residual stress measurement is considered as a nondestructive method (Osman,2000). Residual stress is an important aspect for diamond coating as it may influence not only the adhesion between coating and substrate, but also the maximum thickness to which coatings can be grown, the roughness of the coating as well as the tribological, fracture and fatigue properties (Yongqing et al,2003). The relation between film thickness and residual stress level depends on the process used during film deposition (G.C., 2007).

A new methodology for the evaluation of residual stresses in the polymer matrix of unidirectional polymer matrix composites based on XRD measurements of residual stresses in embedded crystalline inclusions has been recently developed by (Benedikt et al, 2002). The most common methods for measuring residual stresses in coated surfaces are those based on direct measurement of the elastic strains in the film by X-ray diffraction (Pauleau, 2008; Cammarata, 2004; Malzbender, 2002; Zoestbergen, 2002; Perry, 1996; Larsson, 1996; Sue, 1992).

It was found that residual and applied strains transferred to various crystalline inclusions were high enough to be detected by X-ray diffraction. measurement of residual strain by X-ray diffraction (XRD), which is a non-destructive method, has been widely used to investigate the development of residual stress and strain by several researchers (Thanh-Duoc, 2012).

2. OBJECTIVE

The objective of this work is to apply the X-ray diffraction technique to measure and evaluate the residual stresses of pure titanium and anodized titanium.

3. METHODOLOGY

X-ray diffractometer equipped with a computer controlled laboratory diffractor as shown in Figure 1. In Figure 1 the basic geometry of an x-ray diffractometer is shown. X-rays coming from an x-ray source strike the specimen which stabilized by a sample holder. The settings of the X-ray diffractometer are summarized in Table 1.

The experimental data of pure titanium and titanium nanotubes were used for calculated residual stress (Sami et al, 2012). X-rays have wavelengths on the order of a few angstroms (1 Angstrom = 0.1 nm). This is the typical inter-atomic distance in crystalline solids; making X-rays the correct order of magnitude for diffraction of atoms of crystalline materials. A copper target was used, with Cu K α radiation of wave length (λ) = 1.5406Ao , spectra were taken using a power supply of 30 mA and 40 kV. This technique is based on the formation of an interference pattern of waves present in the incident X-ray beam caused by the uniform spacing of the material crystal atoms. A constructive interference pattern will yield peaks of high diffracted intensities and this will occur when the Bragg's Law is satisfied Bragg's Law (Yuri et al, 2012) states that:

$$n\lambda = 2d \sin\theta \dots\dots\dots (1)$$

where n is the order of wavelength, λ is the wavelength of the X-ray source used, d is the lattice spacing and θ is the angle at which the maximum diffracted intensity occurs.

The residual stress was determined by measuring the strain in the crystal lattice of the samples with the assumption that the deformation of the crystal lattice was linearly elastic. The strains were calculated based on the changes in the spacing of the crystal lattice planes (d). The strain can also be expressed in terms of changes in the spacing of the crystal lattice as (Ong-On et al, 2007):

$$\varepsilon = d/d_0 \dots\dots\dots (2)$$

4. RESULTS AND DISCUSSION

The residual stresses of pure titanium was measured by using x-ray diffraction method . The slope obtained from the $\Delta d/d_0$ versus $\sin^2 \theta$ plot in Figures (1 to 3) show square line fitted to data obtained from the x-ray diffraction for pure titanium and anodized titanium at (10V , 30V)and exhibits a regular $\Delta d/d_0$ vs $\sin^2 \theta$ behavior equation (3) (Ong-On et al, 2007). d_0 is the d- spacing before deposition and d is d-spacing after deposition . ν is Poisson's ratio for titanium (0.32) and E is the Young's modulus of titanium (116 GPa) . The slope of the plot was used to calculate residual stress. The in – plane stress was determined using $\sin^2\theta$:

$$\sigma = \text{slope} \times [E/(1+\nu)] \dots\dots\dots (3)$$

Figure 2 shows the tensile stress in the pure titanium . The measured residual stresses in the titanium substrate is 0.88 MPa .

Figure 3 and 4 show the compressive stress dominates in the TiO₂interlayer. The measured residual stresses in the TiO₂interlayer are -6.15 MPa and -8.8 MPa for anodized 10 V and 30 V respectively.

These results seemed to suggest that the annealing process have contributed to some degree of the stress relieving process through recovering, recrystallisation and grain growth of the titanium anodizing .

The relation between film thickness and residual stress level depends on the process used during film deposition (G.C., 2007). The results seemed to suggest that the residual stresses become increasingly compressive with the increase in number of crystallography directions of the material (G.C., 2007). The tribological behavior of thin ceramic coatings is improved by compressive residual stresses parallel to the surface (Kennedy et al, 1990).

5. CONCLUSION

The X-ray diffraction (XRD) is one of the best developed methods available for residual stress determination. It is a non destructive technique. XRD technique uses the distance between crystallographic planes (d-spacing) as a strain gage. This method can only be applied to crystalline, polycrystalline and semi-crystalline materials. When the material is in tension, the d-spacing increases and when the material is in compression. Among the samples have negative values of residual stresses which means the state of residual stress exist in the both samples are

compressive. The diffraction angle 2-theta can be measured experimentally and the d-spacing is then calculated using Bragg's law.

6. REFERENCES

- 1- Osman, A. (2000). Residual Stress Measurement Using X-Ray Diffraction. *Texas A&M University*.
- 2- Yongqing, F., Hejun D., Chang Q. (2003). Interfacial structure, residual stress and adhesion of diamond coatings deposited on titanium. *Thin Solid Films*, 424 ,107–114
- 3- G.C. (2007). Stress and strain in polycrystalline thin films, *Thin Solid Films* 515 ,6654–6664
- 4- Benedikt, B., Predecki, P., Kumosa, L., Rupnowski, P.(2002). KumosaM.Measurement of Residual Stresses in Fiber Reinforced Composites Based on X-Ray Diffraction. International Centre for Diffraction. *Advances in X-ray Analysis*, 247-256.
- 5- Pauleau, Y. (2008). Residual Stresses In DLC Films And Adhesion To Various Substrates, *Tribology of Diamond-Like Carbon Films*.
- 6- Cammarata, R. (2004). Stresses In Thin Films, In: Totten, Liang (Eds.). *Surface Modification And Mechanisms*, Marcel Decker, New York, USA, 17– 29.
- 7- Malzbender, J., Den, J., Balkenende, A. (2002). Measuring mechanical properties of coatings: a methodology applied to nano-particle filled sol–gel coatings on glass. *Mater. Sci. Eng* , 47–103
- 8- Zoestbergen, E., De, J. (2002). Crack Resistance of PVD Coatings: Influence of Surface Treatment Prior To Deposition. *Surface Eng.*, 18 , 283–288
- 9- Perry, A. , Sue J., Martin, P. (1996). Practical Measurements of the Residual Stress In Coatings. *Surface Coatings Technol* , 17–28
- 10- Larsson, M., Hedenquist, P., Hogmark, S. (1996). Deflection Measurements A method to Determine Residual Stress in Thin Hard Coatings on Tool Materials. *Surface Eng* , 43–48
- 11- Sue, J. (1992). X-Ray Elastic Constants and Residual Stress of Textured Titanium Nitride Coating. *Surface Coatings Technol*, 154–159
- Fundamentals and Applications, Springer, New York, USA, 102–136
- 12- Thanh-Duoc, P. , Saden, H., Masood, S., Mahnaz Jahedi. (2012). Finite Element Analysis of Cooling Time and Residual Strains in Cold Spray Deposited Titanium Particles World Academy of Science. *Engineering and Technology*
- 13- Sami, A., Abdulkareem, M., Zamen, K. (2012). Corrosion Behavior of Pure Titanium And Titanium Anodizing In Synthetic Blood Plasma Solution , The First Women National Scientific Conference Under Publuction
- 14- Yuri, S. & Ryan, L. (2012). Spatiotemporal Response of Crystals in X-ray Bragg Diffraction Advanced Photon Source, Argonne National Laboratory, USA
- 15- Ong-on, T., Vittaya, A., Thanusit, B. (2007). X-ray Diffraction Studies on Grain Size of Nanocrystalline Diamond and Residual Stress on Silicon Substrate Prepared By Hot-Filament Chemical Vapor Deposition. *New Diamond and Frontier Carbon Technology*
- 16- Kennedy, F., Tang, L. (1990) Factors Affecting The Sliding Performance of Titanium Nitride Coatings, *Mechanics of Coatings*. Amsterdam, 409–415

Table 1. Settings of the X-ray diffractometer.

Conditions	Settings
X-ray Characteristics	Cu K α
Wavelength (Å)	1.5406
X-ray Tube Current	30 mA
X-ray Tube Voltage	40 kV

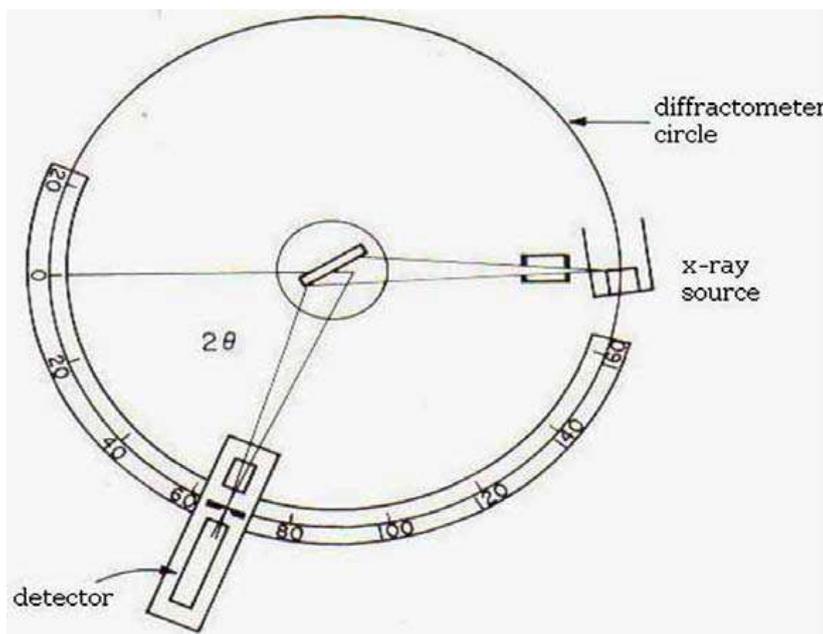


Figure 1. An x-ray

diffractometer scheme [1].

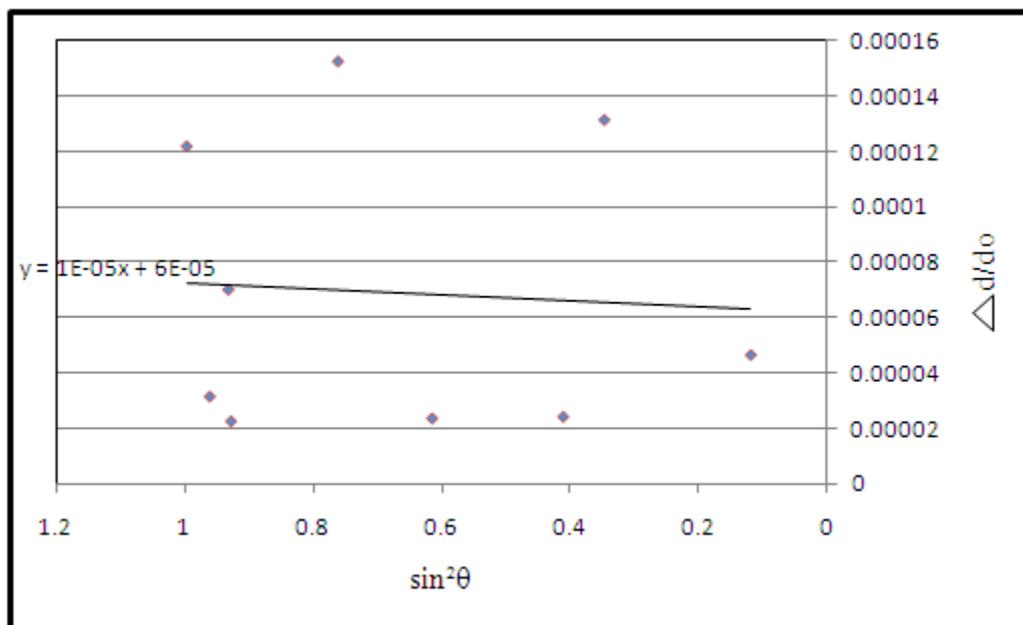


Figure 2. A linear graph of d vs. $\sin^2 \theta$ to diffraction data with positive slope at pure titanium.

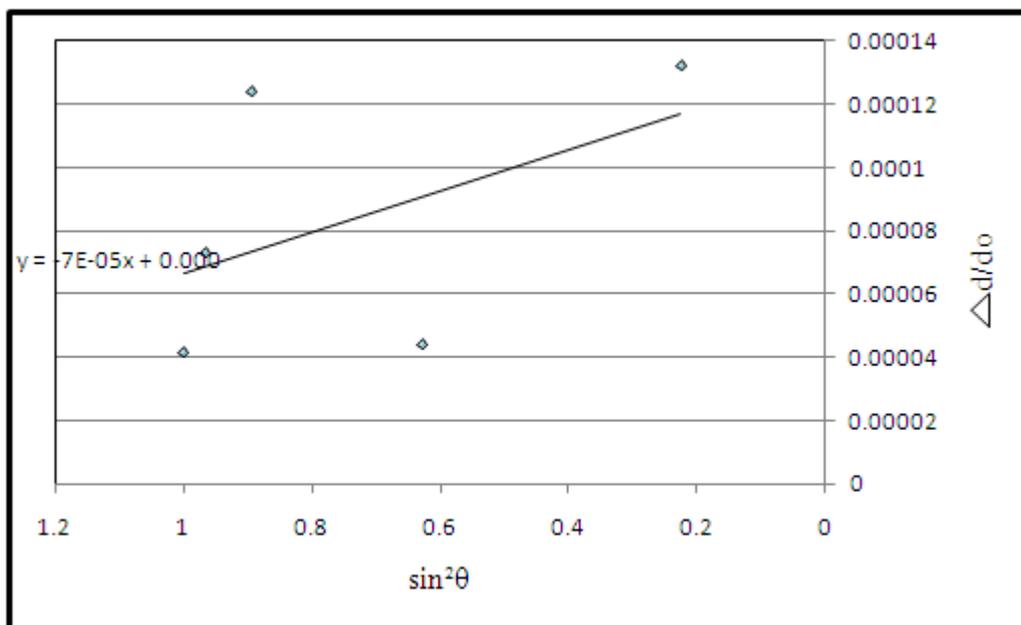


Figure 3. A linear graph of d vs. $\sin^2 \theta$ to diffraction data with a negative slope at anodized 10V.

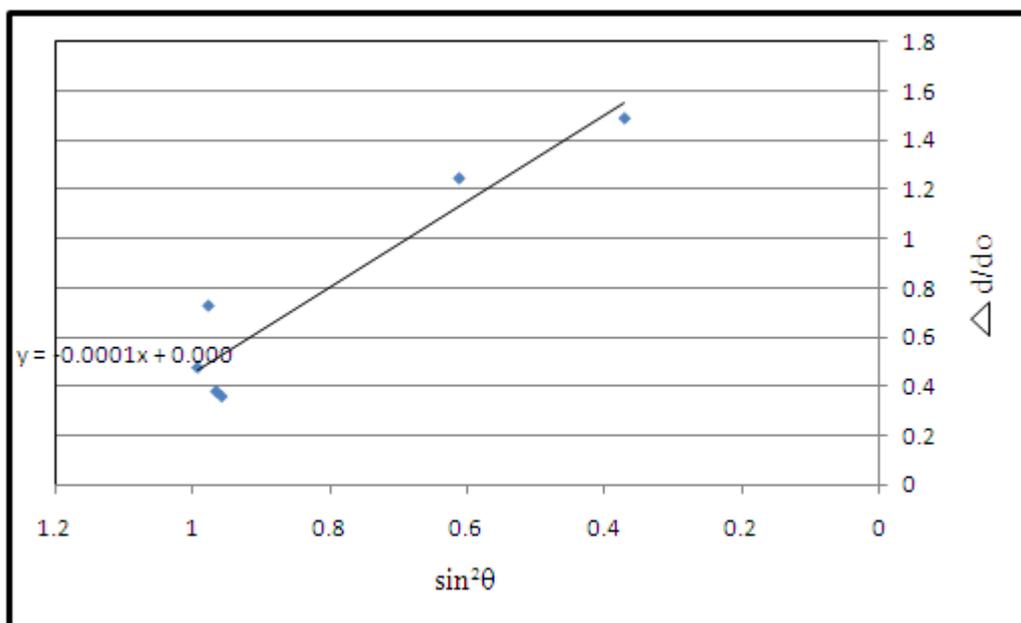


Figure 4. A linear graph of d vs. $\sin^2 \theta$ to diffraction data with a negative slope at anodized 30V.