Residual Stress Measurement of Ti-Metal Samples by Means of XRD with Ti and Cu Radiation

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Abstract: The use of titanium in structural components has been growing for years, especially in highly demanding applications in the aerospace industry where quality control is essential. One of the critical properties is fatigue strength, which is strongly affected by residual stresses. Residual stresses may be an unavoidable by-product of the manufacturing process or intentionally imparted through processes like shot peening. X-ray diffraction (XRD) is commonly used for residual stress measurement. Yet titanium alloys are more difficult to measure than other metals like steels and aluminium alloys. Many titanium alloys have a two-phase microstructure, one of them being hexagonal alpha titanium. Also, the commonly used Cu-radiation generates very strong fluorescence, which results in a low penetration depth. Both reduces measurement quality. Ti-radiation is much less frequently used. It benefits from very low fluorescence and a higher penetration depth. This paper compares XRD residual stress measurements using Cu- and Ti- radiation on two titanium grades: Grade 2 and Grade 5 (Ti6Al4V). The samples from both are in the rolled condition and have been shot peened. The x-ray elastic constants were determined by XRD with Cu- and Ti-radiation and samples and residual stress depth distributions were measured up to 0.5 mm depth.

Introduction

Titanium alloys are widely used in aerospace structures due to their excellent strength to weight ratio. For aerospace applications reliability is critical from which follows that all factors affecting the performance of the components have to be studied and controlled extremely well. One important factor is residual stress, which is always present due to the manufacturing processes and which affects fatigue strength significantly. Titanium alloys are challenging because their machining is difficult due to their high chemical reactivity, poor thermal conductivity, high strength that is maintained at elevated temperatures and low modulus of elasticity [1]. The same factors make welding challenging. Since productivity is always an important issue it is always attempted to minimize the need for machining and welding [2, 3].

For the evaluation of titanium components, reliable, fast and easy residual stress measurement is a key issue. There are several methods for measuring residual stresses nondestructively and destructively. This paper deals with X-ray diffraction, using X-ray instruments specifically designed for residual stress measurement. The measurement accuracy depends on a number of factors, such as grain size, preferred orientation, accessibility of measurement location etc. The X-ray wavelength is given by the X-ray tube chosen. It is preferable to select it so that the 2θ – angle of the diffraction peak analyzed is the highest possible that has sufficient intensity. However, some combinations of material and radiation cause significant fluorescence, which affects the net intensity level and also the effective measurement depth [3]. X-ray diffraction does not measure stress directly, but strain.

The stresses have to be calculated using elastic constants [4]. While X-ray elastic constants (XEC) are the best choice, they are often not available and macroscopic elastic constants determined by mechanical testing are commonly used. It is also possible to calculate elastic constants based on single crystal properties.

CuKα (copper) is the most commonly used radiation to measure titanium alloys. It creates very strong fluorescence. A newer alternative is TiKα radiation (titanium), which has very low fluorescence and nearly twice the penetration depth [5]. The aim of this work is to compare the performance of both of these wavelengths in residual stress measurement. The X-ray elastic constants are also measured with both wavelengths and compared to macroscopic and calculated values.

Experimental procedure

The material used in this work are two commercially available, 3 mm thick, titanium plates, one from commercially pure titanium (Grade 2) and one from two-phase alloy Ti6Al4V (Grade 5). The main phase in both samples is the hexagonal α-phase. Ti6Al4V has a maximum of 10% cubic β-phase [3]. Typical values for some mechanical properties of these alloys are shown in Table 1.

Table 1. Material properties of Grade 2 and 5 titanium alloys [6].

Grade	Ultimate strength [MPa]	Yield strength [MPa]	Elastic modulus [GPa]	Poisson's ratio
Grade 2	241	172	103	0.32
Grade 5	896	827	114	0.32

The samples were shot peened to minimize grain size and preferred orientation effects (Fig. 1).

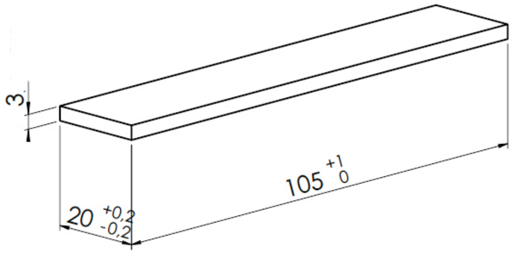

Figure 1. Sample dimensions [mm].

They were loaded on a four-point bending device for the XEC measurements. The bending load was controlled by a calibrated load cell and the load stresses were calculated using Eq. (1):

$$\sigma = \frac{3Fa}{bh^2} \tag{1}$$

in which σ = stress
F = applied force
a = support span
b = width of the specimen
h = thickness of the specimen

Five load steps were used: for Grade 2: 50, 211, 372, 533 and 695 N (corresponding stresses 17, 72, 128, 183 and 283 MPa); for Grade 5: 102, 434, 766, 1099 and 1431 N (stresses 44, 187, 330, 473 and 616 MPa).

X-ray measurements. The X-ray measurements were made using the modified-ψ measurement mode on an Xstress G3 equipped with Mythen detectors. Maximum χ - tilts were ±45°, with four tilts on both sides. The diffraction peak for the family of {213} planes was used with CuKα (Bragg angle 2θ = 139.3°), that for the family of {110} planes with TiKα (Bragg angle 2θ = 137.4°). For Grade 5, the XEC was also measured on a sample not shot peened. This was not possible for the Grade 2 sample due to its large grain size.

The XEC measurements were performed using the method described in the EN15305 [7]. To determine $\varepsilon_{\phi\psi}$, the stress σ^A is calculated for each applied load.

$$\varepsilon_{\phi\psi} = \frac{1}{2}S_2^{hkl} sin^2\theta(\sigma_{11}^R - \sigma_{33}^R - \sigma_{11}^A) sin^2\chi + \frac{1}{2}S_2^{hkl} sin^2\theta\tau_{13}^R sin2\chi + K] \tag{2}$$

where

$$K = S_1^{hkl} Tr(\sigma^R) + \frac{1}{2}S_2^{hkl}(sin^2\theta\sigma_{33}^R + cos^2\theta\, \sigma_{\phi+\frac{\pi}{2}}^A) \tag{3}$$

This represents an elliptical function

$$\varepsilon_{\phi\psi} = a{*}sin^2\chi + b{*}sin2\chi + c \tag{4}$$

where

$$a = \frac{1}{2}S_2^{hkl} sin^2\theta(\sigma_{11}^R - \sigma_{33}^R - \sigma_{11}^A) \tag{5}$$

in which the XEC

$$\frac{1}{2}S_2^{hkl} = \frac{1+\nu}{E} \tag{6}$$

can be evaluated by the least square method. The superscript R with the stress parameters refers to residual stresses, the superscript A to applied stress.

Residual stress depth profiles were measured on the shot peened surfaces. Electropolishing was used to remove material for the various depths. The measurements with CuKα and TiKα were made at exactly the same locations and depths.

Results and analysis

X-ray elastic constant measurements were made starting from high load to low load and back up again. This was performed five times, once for every combination of X-ray radiation, surface condition and material. Typical measurement data are shown in Fig. 2.

The XEC results are summarized in Table 2, which also includes XECs calculated from Young's moduli and Poisson's ratios determined by mechanical testing (macroscopic), XECs predicted from single crystal properties (after Kröner) and some literature data.

All elastic constant values are in the same range, except that the Grade 5 TiKα results are lower by about 20%. There is a systematic trend that plastic deformation (shot peening) reduces the value of the XEC. The same trend has been found by Bahadur et.al. [10] in austenitic steels. The differences between the (213) CuKα and the (110) TiKα values are significant. Some variability in the Grade 5 XECs could be due to its two-phase microstructure. Another, quite probable factor is preferred orientation, which can also affect peak intensity. For a randomly oriented microstructure Imax should decrease moderately as function of sin2ψ or may be nearly constant. With TiKα radiation Imax decreases clearly more than expected and with CuKα it even increases significantly – for the shot peened (Fig. 3) as well as the not shot peened surfaces.

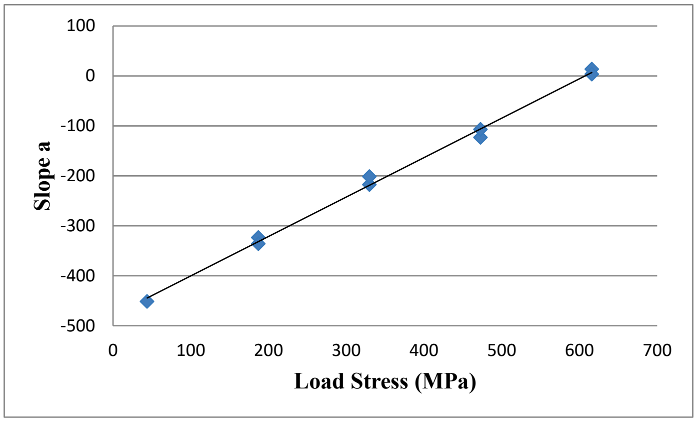

Figure 2. XEC graph for CuKα and Grade 5, measuring from high load to low and back to high. Slope 10.8 ±0.26 10^{-6} MPa^{-1}.*

*Table 2. Measured XEC values (1/2S_2), 10^{-6} MPa^{-1} in italic. Average error of measured XEC's is 0.3*10^{-6} MPa^{-1}*

	Grade 2	Grade 5	Grade 5 (not shot peened)	α-phase, Calculated, Kröner	α-phase, XEC [8]
XEC CuKα (213)	*10.4*	*10.7*	*11.3*	11.7	11.90 **
XEC TiKα (110)	*11.1*	*8.7*	*9.2*	12.0	11.98
Macroscopic*	12.8	11.6			
XEC (213) [9]		10.9/11.9			

*calculated from values in Table 1.
** lattice {302}

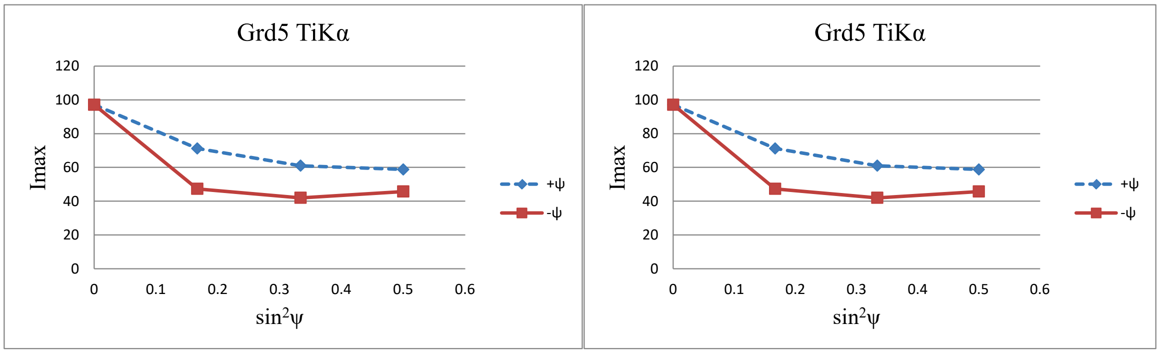

Figure 3. Maximum intensity of diffraction peaks as function of tilt angle for Grade 5 with Tikα (110) and CuKα (213). Examples are from shot peened surfaces.

Residual stress depth distribution. The Grade 5 depth distributions were measured up to 0.5 mm depth and Grade 2 to 0.3 mm depth (Fig. 4). In Grade 2, the grain size became too large at that depth. Stresses were calculated using an XEC of 11.0*10^{-6} MPa^{-1}. The measurements with CuKα and TiKα were made at exactly the same locations, in the longitudinal direction of the samples (Fig. 1). In the

CuKα measurements Mythen detectors were used due to their better sensitivity and a threshold energy capability to decrease the fluorescence radiation [11].

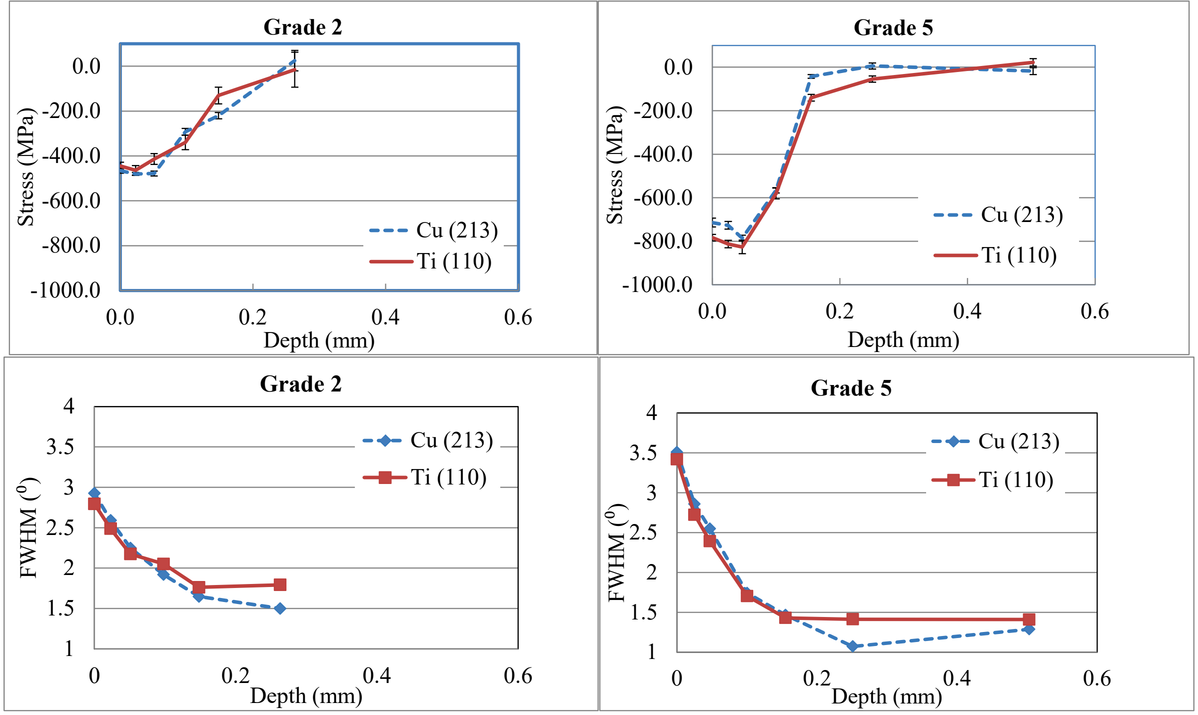

Figure 4. Depth distribution of residual stresses and full width half maximum (FWHM) of shot peened Grade 2 and Grade 5 samples measured by CuKα ($2\theta = 139.3^o$) and TiKα ($2\theta = 137.4^o$).

The CuKα and TiKα measurement results are very close to each other. There is a small difference in the stress levels of Grade 5 (Fig. 4) measured by CuKα and TiKα, where the CuKα values are slightly lower. This correlates with the higher XEC value for CuKα (10.7 vs. 8.7) but the XEC difference is much bigger. Shot peening deformed the material up to about 0.2mm depth, a little bit deeper for Grade 2 than Grade 5. On average, the TiKα measurements have higher error bars compared to CuKα. In the measurements shown in Figure 4, the average error bars were ±15 to ±19 MPa for Grade 5 and ±17 to ±35 MPa for Grade 2. These higher error bars may be related to the multiplicity factor, which is 24 for the {213} peak (CuKα) and only 6 for {110} (TiKα). The higher the multiplicity factor the less sensitive the measurement should be to preferred orientation.

Conclusions

The X-ray elastic constants measured with CuKα radiation for the (213) lattice planes agree reasonably well with values found in literature. Also the unalloyed titanium sample gives similar values for the (110) planes. But the Grade 5 (Ti6Al4V) XEC values measured on (110) planes using TiKα are clearly smaller than other measured and reported values. Slightly larger residual stresses in the (110) measurement support this difference only to a small degree. There is no clear explanation for these low XEC values. It may be related to preferred orientation or the second phase.

The residual stress results with Cu- and Ti-radiations are comparable. Both work equally well provided that the material's strong fluorescence with Cu-radiation can be properly addressed in the data analysis. The benefit of Ti-radiation is its greater penetration depth and the shorter measurement times. On the downside, its diffraction peak has a lower multiplicity factor, which makes the measurement more sensitive to texture.

References

[1] M. Rahman, Y.S. Wong and A.R. Zareena, Machinability of titanium alloys. JSME Ser C 2003; 46(1): 107–115.
http://dx.doi.org/10.1299/jsmec.46.107

[2] D.M. Dimitrov et.al., Residual stress evaluation of a high performance machined pre-formed Ti6Al4V part. Procedia CIRP 45 (2016) 155 – 158.
http://dx.doi.org/10.1016/j.procir.2016.02.049

[3] G. Bruno, B.D. Dunn, Surface and bulk residual stress in Ti6Al4V welded aerospace tanks, Transactions of the ASME Vol. 126, Aug 2004
http://dx.doi.org/10.1115/1.1763932

[4] M.A. Iadicola, T.H. Gnäupel-Herold, Effective X-ray elastic constant measurement for in situ stress measurement of biaxially strained AA5754-O. Materials Science and Engineering A 545 (2012) 168–175
http://dx.doi.org/10.1016/j.msea.2012.02.100

[5] V. Hauk, Structural and Residual Stress Analysis by Nondestructive Methods, Elsevier, 1997 112.

[6] Information on http://www.rtiintl.com/Titanium/RTI-Titanium-Alloy-Guide.pdf

[7] EN 15305:2008 Non-destructive Testing - Test Method for Residual Stress analysis by X-ray Diffraction

[8] V. Hauk, Structural and Residual Stress Analysis by Nondestructive Methods, Elsevier, 1997 135.

[9] I.C. Noyan, J.B. Cohen, Residual Stress Measurement by Diffraction and Interpretation, Springer-Verlag, 1987

[10] A. Bahadur, B. Ravi Kumar and S. Ghosh Chowdhury, Evaluation of changes in X-ray elastic constants and residual stress as a function of cold rolling of austenitic steels, Materials Science and Technology March 2004 Vol. 20
http://dx.doi.org/10.1179/026708304225012170

[11] D. Šišak Jung, L. Suominen, J. Parantainen and C. Hoermann, MYTHEN Detector – Perspectives in Residual Stress Measurements (2014) Adv. Mat. Res. 996, 203-208