

## X-RAY STRESS MEASUREMENTS IN THE INSTITUTE OF AVIATION POSSIBILITIES AND EXAMPLES

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### **Abstract**

*From the point of view of the airplane construction, its fatigue lifetime and exploitation process, the stress states and levels are of crucial importance. The most appropriate experimental methods to determine stress values are diffraction methods with different radiation type employed. These methods allow the determination of the elastic lattice deformation and distortion (effectively the stress state) from the displacement and broadening of the diffraction peak. Diffraction methods are widely known as the experimental methods for determining not only the stress values but also the elastic properties of polycrystalline materials (also of all alloys types used in the aerospace industry). The advantages of diffraction experiments result from their non-destructive character and the possibility to obtain absolute values of stresses in different phases of each type of crystal material (the measurements are performed selectively only for crystallites contributing to the measured diffraction peak, i.e. for the grains having lattice orientations for which the Bragg condition is fulfilled). In the frame of this work, the laboratory possibilities of the Institute of Aviation in this area are presented as well as the exemplary results of stress measurements performed there.*

**Keywords:** X-ray diffraction, residual stress measurements, fatigue

### **1. X-ray diffraction and its application in stress values estimation**

Residual stress measurement by diffraction methods, unlike other stress measurement methods, allow to obtain the information on absolute values, i.e. information on stress values in relation to the unstrained material, not to initial state of the specimen (like in case of strain gages application). This method consists in using the phenomenon of X radiation diffraction on the nodes of the crystal lattice. Mathematically, this phenomenon is described by Bragg's law.

#### **1.1. Bragg's law and the principles of stress measurement with diffraction methods**

The real structure of a crystalline material can be approximated by a system of atoms periodically distributed in three dimensions. Such a regular periodic lattice may be a source of constructive or destructive interference of radiation but only if the wavelength of the radiation used ( $\lambda$ ) is close to the distance between the planes of the crystal lattice  $d$ . Bragg's law was a mathematical description of an experiment carried out by Max von Laue in 1912, which consisted in overexposing copper sulphate crystals to X-rays. The mathematical formula of this law is [2]:

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

where:

$n$  – natural number,

$\lambda$  – the wavelength of the X radiation used,

$d$  – interplanar spacing of examined material,

$\theta$  – Bragg's angle, the angle between the lattice plane and the incident beam direction.

Analysing the above equation (1), it can be concluded that the angle  $\theta$  will change if the inter-plane distances of the material change, e.g. due to its deformation. Therefore, if we compare the diffractograms resulting from the diffraction experiment for a strained material with that for a strain-free material, it will be possible to calculate the strain to which the material is subjected by comparing the values of Bragg's angle  $\theta$ . In other words, any change in the material's interplanar distances will shift the position of the diffraction peaks. More specifically, if the material is compressed, the interplanar spacings between surfaces are reduced and the Bragg's angle will increase (the diffraction peaks on the diffractogram will be moved to the right). Each time when the material is subjected to tensile forces, the diffraction angles are reduced, thus moving the diffraction peaks to the left.

In case of stress measurements by X-ray diffraction, it is necessary to assume that the stress value in the direction perpendicular to the tested surface is equal to 0. Such an assumption is justified because X-ray measurement is performed for the surface layer of about a dozen micrometres. The deformation value can be calculated from the following equation [2, 3]:

$$\varepsilon_{\psi} = \frac{d_{\varphi\psi} - d_0}{d_0}. \quad (2)$$

This equation uses the information about the values of the interplanar spacings in the unstressed material  $d_0$  and in the specimen examined  $d_{\varphi\psi}$ .  $\varphi$  and  $\psi$  stand for angles between the incident beam and some directions with respect to the specimen geometry:  $\varphi$  is an angle between the projection of the incident beam on the specimen surface and the some distinguished direction of the specimen,  $\psi$  is an angle between the incident beam and its projection on the specimen surface. Both of these values,  $d_0$  and  $d_{\varphi\psi}$ , are calculated by applying the Bragg's law to the data of the diffraction experiment. In order to convert strain values into stress, Hooke's law and Young's modulus ( $E$ ) should be used, while stress value in a perpendicular direction, assuming plane stress state, can be determined using Poisson's coefficient ( $\nu$ ).

Using the isotropic theory of elasticity it is possible to determine the value of deformation, in direction defined by the angles  $\varphi$  and  $\psi$ :

$$\varepsilon_{\varphi\psi} = \frac{1+\nu}{E} (\sigma_1 \cos^2 \varphi + \sigma_2 \sin^2 \varphi) \sin^2 \psi - \frac{\nu}{E} (\sigma_1 + \sigma_2). \quad (3)$$

This equation can be transformed into a form from which the stress value in direction  $\phi$  can be obtained:

$$\sigma_{\varphi} = \frac{E}{(1+\nu) \sin^2 \psi} \left( \frac{d_{\psi} - d_0}{d_0} \right). \quad (4)$$

The most common in engineering technique are the measurements using so-called  $\sin^2 \psi$  method employing the above equations in the analysis of experimental data. This method uses the information on interplanar spacings measured at different angles of the incident radiation beam with respect to the surface of the sample ( $\psi$ ). The analysis consists on the linear regression method application to the experimental data illustrated as a relation of the distance between planes  $d$  and  $\sin^2 \psi$  (see Fig. 1).

The slope of the fitted to the experimental data line is the value of the stress in the direction specified by the angle  $\varphi$ . This result depends on the elastic properties of the examined material. For the analysis, the macroscopic values describing the elastic properties can be used but the better solution is to determine the values of so-called X-ray elastic constants for given  $hkl$  directions of the crystal lattice by the *in situ* bending test [4, 5]. The procedure of determining these values is described by the ASTM E-1426-94 standard.

In more complex cases, other methods are used. Such situations include, among others, the coexistence of shear stresses, which is associated with the appearance of the so-called *splitting* on  $d$  vs.  $\sin^2 \psi$ . Another situation is the coexistence of 2nd order stresses – in such a situation the oscillations occur around a line fitted to the experimental points.

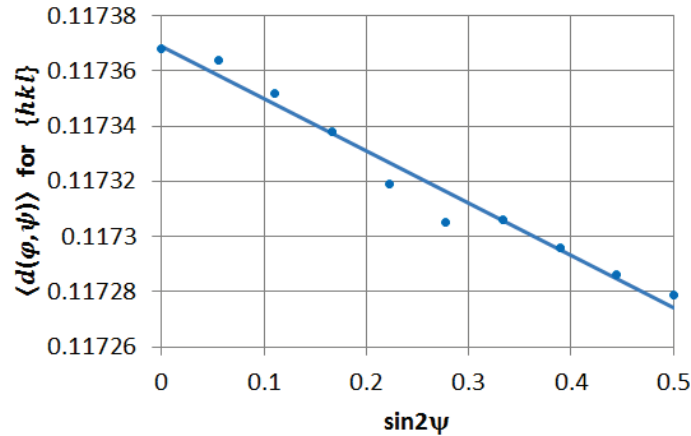


Fig. 1. Typical  $\sin^2\psi$  plot obtained for 17-4PH stainless steel specimen [3]

Another important aspect of X-ray stress measurements is the fact that the intensity of the incident X-ray beam decreases with the depth into the material. The X-ray diffraction method enables the measurements in the surface layer of the material and sometimes it is important to know how thick the layer of material examined is. This value depends on the type of radiation used, its angle of incidence, and the coefficient of X-ray absorption ( $\mu$ ) for the type of radiation used. The thickness of the effective layer can be estimated using the equation below [2]:

$$x = \frac{\ln\left[\frac{1}{1-G_x}\right]}{\mu\left[\frac{1}{\sin(\theta+\psi)} + \frac{1}{\sin(\theta-\psi)}\right]}. \quad (5)$$

$G_x$  stands for total intensity diffracted by a finite layer expressed as a fraction of the total diffracted intensity [2].

## 2. The Institute of Aviation X-ray diffraction laboratory, its equipment and possibilities

The Laboratory of X-ray Diffraction in the Institute of Aviation in Warsaw, has at its disposal the X-ray diffractometer produced by Stresstech Oy (Finland), Xstress3000 G2/G2R. The diffractometer is equipped with X-ray tubes of 4 types serving the stress measurements of different material types, i.e. chromium (dedicated to aluminium alloys and steels), manganese (e.g. Inconel, austenitic steel), titanium (e.g. titanium alloys) and vanadium (e.g. austenite and copper). In Xstress3000, diffractometer the diffracted beam is collected by two symmetrically positioned CCD detectors, which enable the collection of one  $hkl$  reflection at once. The radiation of incident beam can be collimated by collimators of different sizes from 0.5 mm to 5 mm. The diffractometer applies the standard  $\sin^2\psi$  method and the range of the  $\psi$  angles is from  $-45^\circ$  to  $+45^\circ$  while it can be extended by an application of tilting grip. Another advantage of the diffractometer is the X-Y system allowing the automated mapping of a surface examined; it makes possible to evaluate the stress map of the surface with a micrometre accuracy. The measurement with this equipment is supported by the Xtronic software, which serves the aquired data analysis. The software enables the fitting of different curves to the peak profiles. Another advantage of the X-ray Diffraction Laboratory in the Institute of Aviation is the electro polishing machine Kristall 650 which enables the in-depth gradients measurements.

The procedures of the laboratory meet the international standards of stress determination with diffraction methods, i.e. PN-EN 15305:2008 (Non-destructive Testing - Test Method for Residual Stress analysis by X-ray Diffraction), SAE J784a (Residual Stress Measurement by X-Ray Diffraction) and ASTM E915-16 (Standard Test Method for Verifying the Alignment of X-Ray Diffraction Instrumentation for Residual Stress Measurement).

### 3. Exemplary stress measurements with X-ray diffraction

#### 3.1. Ferritic steel

The first example of X-ray stress measurements are those on specimen prepared for the in-depth stress gradient measurements. It was made of 1.4923 high strength chromium stainless steel with molybdenum in addition which common applications are pressure vessels and boilers, aerospace, reactor engineering and turbine. The chemical composition of this steel is presented in Tab. 1.

Tab. 1. The chemical composition of 1.4923 stainless steel [6]

C	Si	Mn	P	S
0.18-0.24%	≤ 0.5%	≤ 0.4-0.9%	≤ 0.025%	≤ 0.015%
Cr	Mo	Ni	V	
11-12.5%	0.8-1.2%	0.3-0.8%	0.25-0.35%	

The mechanical and physical properties are presented in Tab. 2.

Tab. 2. Mechanical and physical properties of 1.4923 stainless steel [6]

$R_p$ [N/mm <sup>2</sup> ]	600	Density $\rho$ [g/cm <sup>3</sup> ]	7.7
$R_m$ [N/mm <sup>2</sup> ]	800-950	Specific heat capacity [J/kg · K]	460
$A_5$ [%]	14	Thermal conductivity [W/m · K]	27
$E$ [kN/mm <sup>2</sup> ]	200	Electrical resistivity [ $\Omega \cdot \text{mm}^2/\text{m}$ ]	0.6

In case of this material the measurements were performed twice for a specimen surface after grinding and after the electrologically remove of 0.2 mm layer. The photo of the specimen for both stages are presented on Fig. 2a and b.

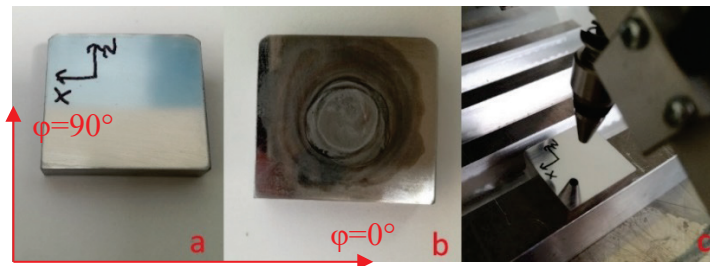


Fig. 2. The specimen made of 1.4923 stainless steel: a) after grinding, b) after electro polishing, c) during the measurement

The measurement was performed in both cases with a use of 3 mm collimator. The radiation used became from the chromium X-ray tube. Radiation wavelength was  $\lambda_{K\alpha_1} = 2.29107\text{\AA}$ . The ferritic phase examined in this experiment had a body centred cubic crystal cells and the measurement concerned the  $\{211\}$  planes for which the Bragg's angle was  $2\theta = 156.4^\circ$ . The measurement was performed for 10 tilts for both sides of the goniometer while the time of the measurement for one tilt was 15 s. The stress values were determined for three directions with respect to the specimen geometry, i.e.  $\varphi = 0$ ,  $\varphi = 45^\circ$  and  $\varphi = 90^\circ$ . The calculations and data treatment were performed with an assumption of macroscopic Young modulus equal to 200 GPa and Poisson's ratio  $\nu = 0.3$ . The background of the diffracted signal was linearly subtracted and the peaks shape was fitted with cross-correlation method [1]. The example of resulting diffraction peaks are presented on the Fig. 3.

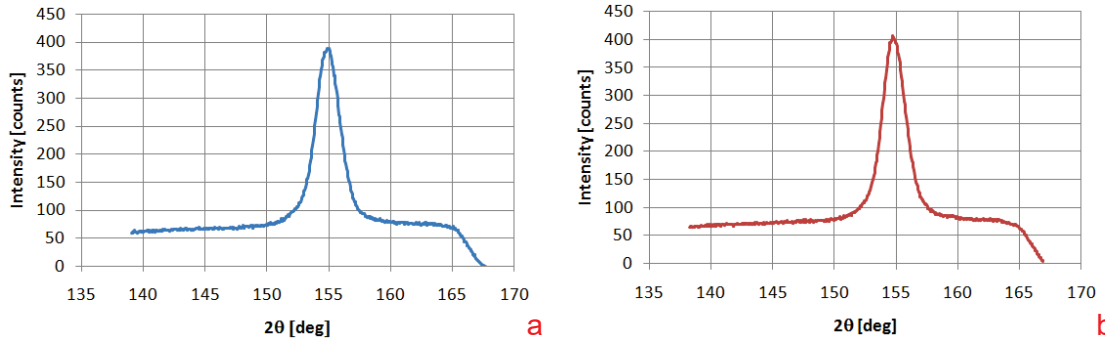


Fig. 3. Diffraction peaks for 1.4923 stainless steel resulting from the X-ray diffraction experiment

The linear regression fitting and stress values in defined  $\varphi$  directions are presented in Figs. 4 and 5 and in Tab. 3 and 4.

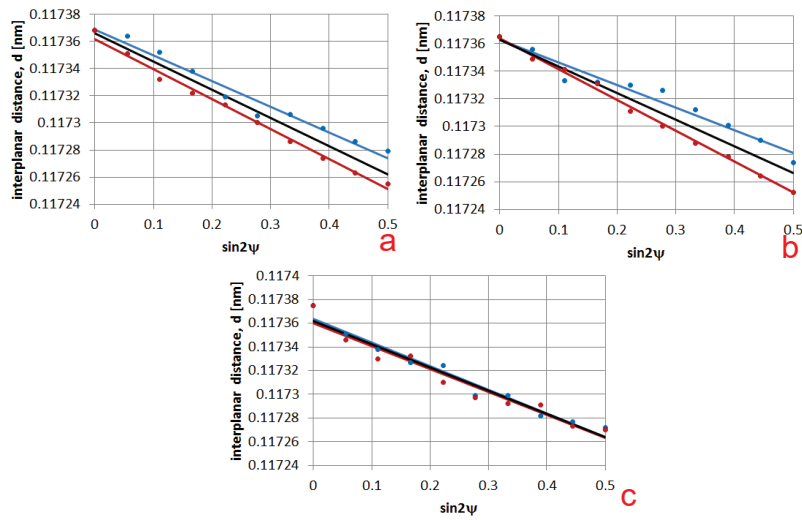


Fig. 4 The  $d$  vs.  $\sin^2\psi$  graphs for the 1.4923 specimen after grinding in three directions a)  $\varphi=0^\circ$  b)  $\varphi=45^\circ$  and c)  $\varphi=90^\circ$

Tab. 3. Stress measurement results for the 1.4923 specimen after grinding

$\varphi$ [°]	$\sigma$ [MPa]	$\Delta\sigma$ [MPa]	FWHM [°]	$\Delta$ FWHM [°]
0	-284.3	9.1	2.40	0.11
45	-267.3	9.2	2.39	0.11
90	-270.8	14.4	2.41	0.10

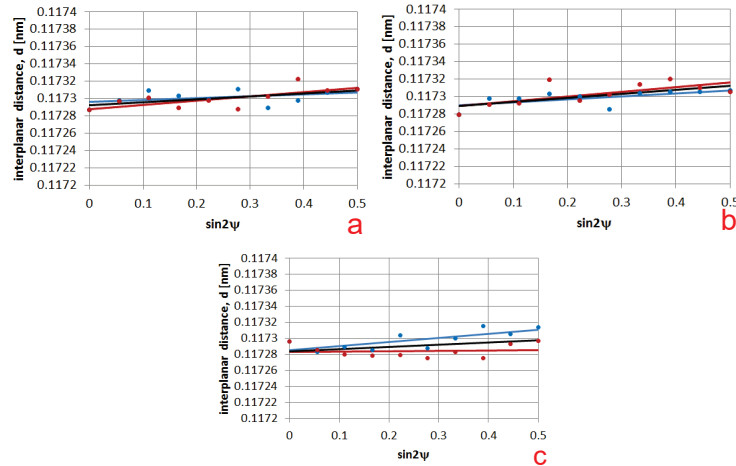


Fig. 5. The  $d$  vs.  $\sin^2\psi$  graphs for the 1.4923 specimen after electropolishing in three directions a)  $\varphi=0^\circ$  b)  $\varphi=45^\circ$  and c)  $\varphi=90^\circ$

Tab. 4. Stress measurement results for the 1.4923 specimen after electro polishing

$\varphi$ [°]	$\sigma$ [MPa]	$\Delta\sigma$ [MPa]	FWHM [°]	$\Delta$ FWHM [°]
0	35.7	15.7	2.10	0.10
45	51.9	16.4	2.07	0.06
90	29.6	19.1	2.08	0.09

The results show the precisely determined stress values for both: the specimen after grinding and after electropolishing while values for underneath levels of the material are significantly different. The stresses on the surface of the specimen are compressive and its value in all three directions are comparable while the values after the remove of 0.2 mm layer are tensile and not as high those on the surface after grinding.

### 3.2. Ti/TiC composite

The interesting diffraction image was obtained for Ti/TiC composite. The specimen used in the experiment is presented on the Fig. 6 below.

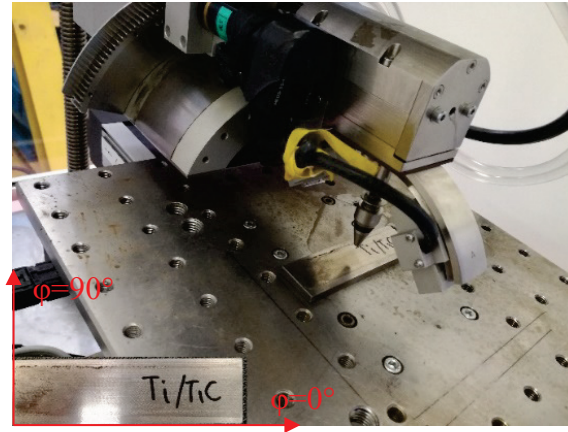


Fig. 6. The Ti/TiC specimen

As the X-ray diffractometry is a method which enable the selectively measurements (i.e. it differentiate the phases of the alloy) the strain and stresses on the interesting phases can be estimated. The example of measurements of this kind are that performed for Ti/TiC composite. The diffraction peaks for Ti/TiC composite are presented on Fig. 7. On the obtained diffractograms two different diffraction peaks, having different intensities can be observed.

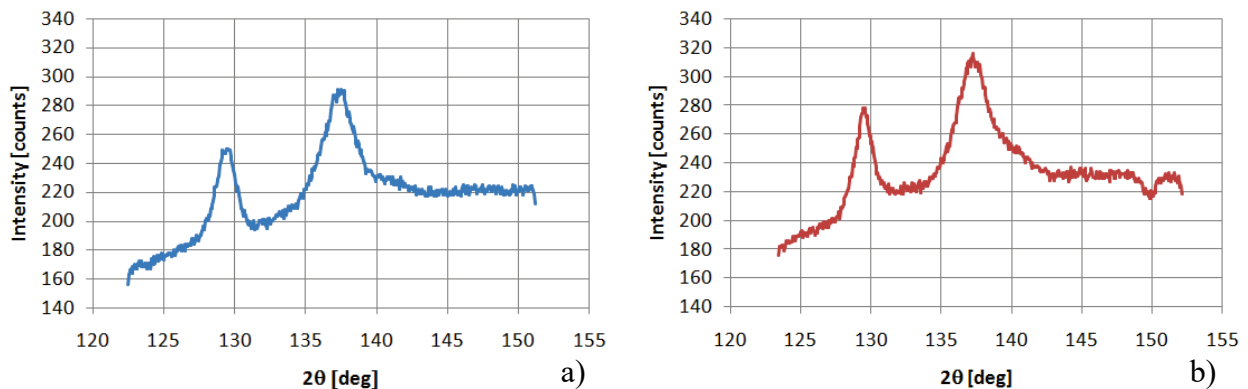


Fig. 7. Diffraction peaks for Ti/TiC composite resulting from the X-ray diffraction experiment

The measurement in this case was performed with the radiation of titanium tube application, which wavelength of  $K_{\alpha_1}$  radiation is 2.7485 Å. For this type of radiation the reflection from the hexagonal titanium cells appears for about  $2\Theta=137.4^\circ$ . The exposure time for one tilt lasted 60 s and the average intensity of diffracted beams was about 102 points what is a sufficient value for stress measurement analyses. The measurement was performed for 7 tilt values for positive ranges of tilts and adequately for negative ranges. The stress values were determined in three directions with respect to the specimen, i.e.  $\varphi=0^\circ$ ,  $\varphi=45^\circ$  and  $\varphi=90^\circ$ . The X radiation spot on the specimen size in this case was 3 mm. The data treatment and stress values calculation assumed the linear background subtraction, the cross correlation method for peak shape fitting, the Poisson's ratio value of 0.36 and the Young modulus equal to 120.2GPa.

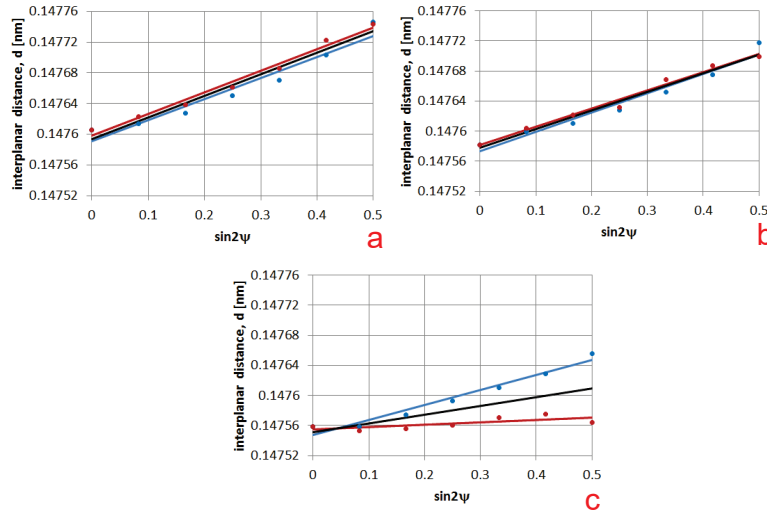


Fig. 8. The  $d$  vs.  $\sin^2\psi$  graphs for the Ti/TiC composite in three directions: a)  $\varphi = 0^\circ$ , b)  $\varphi = 45^\circ$ , c)  $\varphi = 90^\circ$

The comparison of linear regression to the experimental data in three direction with respect to the specimen geometry is presented on the Fig. 8. The regression line is very closely fitted to the experimental data what can be read as a lack of the stresses of the other types in the examined specimen. Additionally some splitting between the data resulting from tilting in “positive” and “negative” direction of the goniometer arc can be observed in direction  $\varphi = 90^\circ$ . It can be associated with the shear stresses in this direction – its value can be calculated as well. The full results of this experiment are presented in Tab. 5.

Tab. 5. Stress measurement results for Ti/TiC composite

$\varphi=[^\circ]$	$\sigma$ [MPa]	$\Delta\sigma$ [MPa]	FWHM [ $^\circ$ ]	$\Delta$ FWHM [ $^\circ$ ]
0	192.4	11.7	3.77	0.35
45	172.1	9.6	3.78	0.35
90	79.6	8.0	3.77	0.31

On the surface of the Ti/TiC specimen, the tensile stresses are present while the measurement concerned the titanium phase of the whole composite. The interesting result could be obtained when the strains of TiC phase would be considered what a next stage of the data analysis is.

#### 4. Summary – the perspectives of the X-ray diffraction laboratory development

The article shows the possibilities of the stress measurements with diffraction methods in the Institute of Aviation in Warsaw. As it was shown the application of the X-ray diffraction phenomenon, have very strong physical principles what makes it reliable. The method is well known worldwide and standardised. Moreover, it enables the estimation of absolute stress values.

In connection with this, the method should be considered when the knowledge on the stress state is necessary. In addition, this method is only one stress measurements method, which enables the subsurface absolute stress values estimation applying the electro polishing procedure.

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