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MULTICONVOLUTIONAL APPROACH FOR UNCERTAINTY ESTIMATION OF THE RESIDUAL STRESSES MEASURED BY XRD SIN$^2\psi$ METHOD

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The paper addresses the multiconvolutional method for estimating measurement uncertainty of the residual mechanical stresses into gold thin layers using the "sin2(ψ)" X-ray diffraction method. The distribution of the mean variable is calculated as a multiconvolution of the probability distribution function of the measurand. The assigned values to the measurand and to its standard uncertainty are the expectance and standard deviation of the distribution of the mean, respectivelly . The same, the expanded uncertainty U(95%) is calculated based on the distribution of the mean variable. If an uniform distribution is assigned to the measurand then the hypotheses number regarding sthochastic behaviour of the measurand reaches its minimum. The multiconvolutional approavch complies with the requirements of the SR EN ISO/IEC 17025 [1] and SR EN ISO Guide 98-3 [2] standards. The paper presents a case study on the advantages of the proposed method compared to the GUM method [2] in the field of residual stress appraisal using the "sin2 (ψ) " method.

Keywords: residual stresses, measurement uncertainty, multiconvolution, uniform probability density distribution, confidence level.

1. Introduction

According to EN ISO/CEI 17025 standard, any test result has to be presented together with its measurement uncertainty (MU) [1] otherwise the result cannot be compared or cannot be used for establishing the conformity of the measurand with a range criterion. The development of new methods for residual stress (RS) measurement or improving the existing ones are not only scientific challenges, but it is a permanent demand generated by many applications where RS plays a critical role as aeronautics, railway welding, bearings, gears, coatings, thin films for electronics, heat treating of steel product etc[3-6]. Depending on application, the internal mechanical stresses are measured by different method as strain gauges, holography, photo elasticity, Moiré fringes, electrical tensometry, laser, ultrasonic, X-ray diffraction, eddy current methods[3-9]. The "$\sin^2(\psi)$" method is the most used worldwide X-ray diffraction method for RS measurement [4, 10, 11]. The exactness of this method depends on the X-ray diffractometer

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performances, but on other many influence factors of the uncertainty budget[4,12-16]. The outcome of the X-ray “$\sin^2(\psi)$” method is a diffraction line i.e. $I(2\theta)$. The location ($2\theta_m$) of the maximum value (I_m) of the diffracted intensity is correlated with the RS value denoted σ. The uncertainty budget of σ consists of equipment factors (diffractometer alignment, divergence of the diffraction geometry, fluctuation of the counting chain etc.), specimen factors (homogeneity of the crystallite shape and dimensions, structural defects, preparation, placement on goniometric stage etc), operational factors (specimen handling, parameter setting of the equipment etc), environmental factors (temperature, vibrations, oxidation). An uncertainty budget of many factors that are not entirely known, partially, or even unknown makes very difficult the MU appraisal or even impossible to be assessed. In this direction, the multiconvolutional approach is of great help because it does not require any knowledge about each influence factor as Guide to the Expression at Uncertainty in Measurement (GUM) standard method requires, but only the range of measurand variation.

2. Theoretical bases of the multiconvolutional approach for MU estimation

In measurement practice, the **probability density function of a uniform variable** ***(updf)*** is assigned to an experimental measurand when there is no information or experimental evidences that its values have a clustering tendency. This is the case of the “$\sin^2(\psi)$” method. In such cases, the experimentalists have to consider that the **probability density function** *(*pdf) assigned to the measurand is of the uniform type denoted as updf. The pdf of a uniform variable X is:

$$f(x) = \begin{cases} 1/(b-a); & x \in [a,b] \\ 0; & x \notin [a,b] \end{cases} \tag{1}$$

where $[a,b]$ is the range variability of X

Any updf can be standardised by a coordinate transformation [17]. Based on the above consideration this paper addresses only standard updfs.

$$f(x) = \begin{cases} 1/2; & x \in [-1,+1] \\ 0; & x \notin [-1,+1] \end{cases} \tag{2}$$

The main parameters of an updf are: $\mu = \frac{a+b}{2}$; $SD = \frac{b-a}{\sqrt{3}}$; $\mu_{2k+1} = 0$ while for a standard *updf* are: $\mu = 0$; $SD = \sqrt{3}/3$.

The sum of two random uniform variables X_1, X_2, denoted $Y_2=X_1+X_2$, is a random variable whose *pdf* is:

$$f_{Y_2}(y) = \int_{-\infty}^{+\infty} f_{X_1}(u) f_{X_2}(y-u)du = f_{X_1} \otimes f_{X_2}(y) \tag{3}$$

where: $f_{Y_2}(y)$ is the convolution of the f_{X_1} and f_{X_2}.

The convolution of two functions is a mathematical operator which has specific properties as: commutative and associative, but the most important property lies in the fact that the Fourier transform of $f_{X_1} \otimes f_{X_2}$ is the product of the Fourier transforms of the respective functions [17, 18].

Based on Eq. (3) there were derived to that the *pdf* of the variable $Y_n(y) = X_1 + X_2 + ... + X_n$ is:

$$F_{Y_n}(y) = f_{X_1} \otimes f_{X_2} \otimes ... \otimes f_{X_n}(y) \tag{4}$$

where: f_{X_i}; $i = 1 - n$ are the *pdfs* of the X_i variable.

The variable $\overline{\overline{X}}_n$, assigned to the mean of n numerical results obtained in repeatability conditions, also called as sample mean variable, is the typical variable to which the linear compound variables theory is applied. Thus, the $\overline{\overline{X}}_n$ has the expression:

$$\overline{\overline{X}}_n = \frac{1}{n}(X_1 + X_2 + ... + X_n) = \frac{1}{n}Y_n \tag{5}$$

where X_i, $i = \overline{1,n}$, are the variables assigned to each measurement.

The *pdf* of $\overline{\overline{X}}_n$ is:

$$f_{\overline{\overline{X}}_n}(\overline{x}) = n \cdot f_{Y_n}(\overline{\overline{x}} \cdot n) = n \cdot f_X^{\otimes n}(n \cdot \overline{\overline{x}}) \tag{6}$$

If $f_X(x)$ is known, then the experimental mean distribution can be calculated, and subsequently, the dispersion of the experimental mean around the conventional true mean μ of X can be estimated as well.

The authors have derived the expressions of $f_{(x)}^{\otimes n}$, n=1- - 5, (Eq.6). The expressions of the n-convolved standard *updfs* are difficult to be calculated for n>5, but one can get help using the general form of a sum of *n* identical standard uniform variables given by Renyi[19]:

$$f_{(x)}^{\otimes n} = \begin{cases} \dfrac{1}{(n-1)!2^n} \displaystyle\sum_{i=0}^{\hat{n}(n,x)} (-1)^1 \cdot C_n^i (x+n-2i)^{n-1}; & -n \le x \le n \\ 0 & ;\ otherwise \end{cases} \tag{7}$$

where $\hat{n}(n,x) = \left[\dfrac{x+n}{2}\right]$ is the largest integer less than $\dfrac{x+n}{2}$

The mathematical expression of the pdf of the mean of the 5 uniform distributed results obtained in repetitive or reproductive condition, denoted $f_{M5}(m) = 5 f_{(5m)}^{\otimes 5}$ is:

$$f_{M5}(m) = \begin{cases} \dfrac{10}{2^5 \cdot 4!}\left(115 - 6 \cdot 5^3 m^2 + 3 \cdot 5^4 \cdot m^4\right) & ; |m| < \dfrac{1}{5} \\ \dfrac{20}{2^5 \cdot 4!}\left(55 - 50|m| - 6 \cdot 5^3 m^2 + 2 \cdot 5^4 \cdot |m|^3 - 5^4 \cdot m^4\right) & ; \dfrac{1}{5} \le |m| < \dfrac{3}{5} \\ \dfrac{5^5}{2^5 \cdot 4!}(1-|m|)^4 & ; \dfrac{3}{5} \le |m| \le 1 \\ 0 & ; |m| > 1 \end{cases} \tag{8}$$

The graph shape of the n-fold convolved standard *updf*s becomes as blunt as the n increases (Fig. 1).

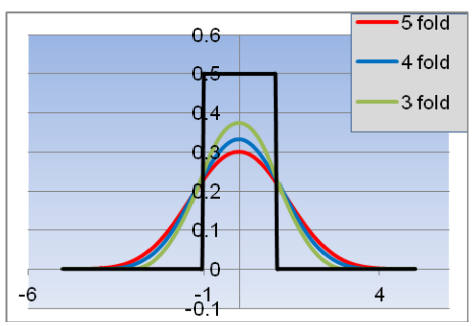

Fig. 1. The graphs of the n-fold convolved standard *updf* for $n = 1,\ldots,5$.

The *pdfs* of the sample means for $n = 1,\ldots,5$ repetitive results were derived (fig. 2). On the contrary to the n-fold convolved function, the *pdf* profile of the mean variable becomes as sharper as the n increases which is shown in fig. 2. A special attention is drawn to $f_{M5}(m)$ because it is appropriate for the most tests where the standard recommends five reproductive measurements [20].

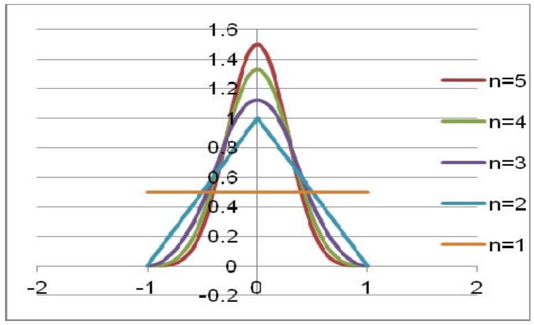

Fig. 2. The graphs of the mean variables for n=1,...,5

Based on the f_{M5} pdf there was calculated the probability that the standardized mean variable of five repeated tests lies in the interval $[-0.2;+0.2]$ i.e. $P(|m|<0.2)\cong 54.3\%$. The probability that the M5 lies in the range $[-0.6;-0.2]\cup[0.2;0.6]$ is $P(0.2\le|x|<0.6)=42.1\%$ while $P(0.6\le|x|<1)=1.8\%$. On the other side, the probability that the mean lies in the interval $[-0.1;0.1]$ is about 32.2% while in $(-0.2;-0.1]\cup[0.1;0.2)$ is 22.1%. Thus, the probability that the mean M5 departs from zero with 0.6 of its pdf half width is about 98.23%. The interval $[-0.47;+0.47]$ could be assigned to the classical 95% confidence level i.e. the conventional true value of the measurand may lie in the interval $m[-0.47a;m+0.47a]$ [m-0.47a; m+0.47a], where a is the half-width of the updf. Thus, expanded uncertainty in the case of 95% confidence level is $U(95\%)=0.47a$.

If one adopts the GUM approach then he has to calculate the combined uncertainty (u_c) and to consider expanded uncertainty having 95 % confidence level as $U(95\%)=2u_c$ [21].

3. Experiments

There were investigated residual stresses located in the Au layer surface deposited on the Si (111) substrate by a conventional electrolytic process used in electronic circuit production.

The stress originates in thin films due to substrate-film differences in thermal expansion, or due to epitaxial mismatch. RS have been calculated using the well known equation of the $\sin^2\psi$ method [22]:

$$\sigma=-\frac{E}{2(1+\nu)}ctg\theta_0\frac{\pi}{180}\frac{\partial(2\theta)}{\partial(\sin^2\psi)} \tag{9}$$

where E is the Young Modulus, ν is the Poisson ratio, $2\theta_0$ is the Bragg angle at ψ=0 and 2θ is the centered position for ψ tilting.

Eq.(9) shows that σ is proportional to the slope of the 2θ vs. $\sin^2\psi$ plot.

According to the metrological practice, the coordinate 2θ of the diffraction peak is estimated for 3-6 tilts of ψ.

The XRD patterns were achieved using a DRON 3 diffractometer which was operated at U=40kV, I=35 mA. The radiation emitted by a Mo anode was filtered with a Zr filter to eliminate K_β and a large part of $K_{\alpha2}$ characteristic lines. The specimens were mounted in the centre of a standard goniometer with Bragg-Brentano geometry. The diffracted radiation was detected with a scintillation based chain detector using a narrow discrimination window for the pulse height. The setting of the tilting angle was done manually using the goniometer θ-2θ adjustment facilities. The specimen stage has been rotated during the pattern acquisition to average the inhomogeneity of the stresses in the sample. The X-ray diffraction patterns obtained in aforementioned conditions on the specimens Au-1, Au-2 and Au-3 are given in Fig. 3

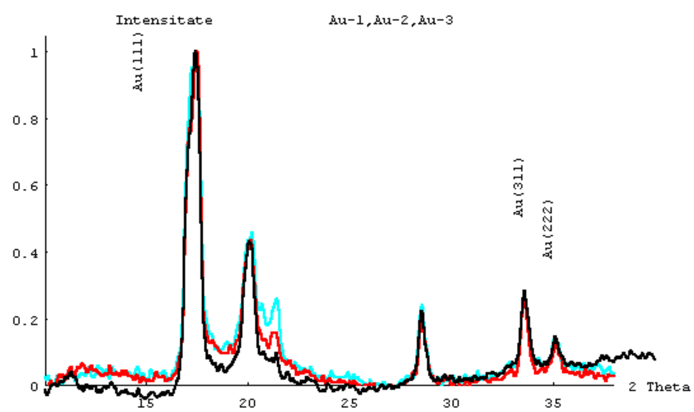

Fig. 3. The X-ray diffraction patterns of the Au-1, Au-2 and Au-3 specimens

The (311) peaks were used for estimating the residual stresses. Therefore, the (311) lines were detailed by decreasing goniometric step from 0.05 to 0.02°. This approach increases the roughness of the X-ray diffraction profile as is shown in Fig. 4

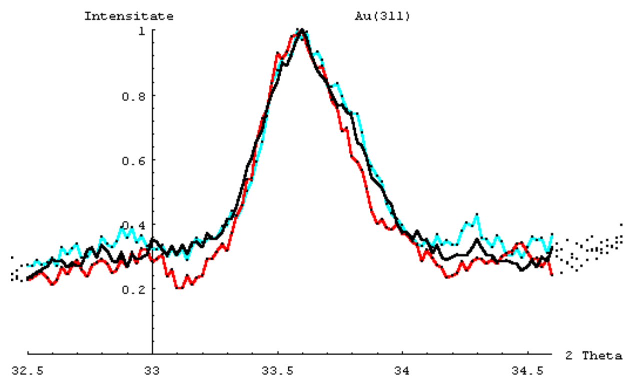

Fig. 4. The detailed (311) line profiles of the Au-1, Au-2 and Au-3 specimens for ψ=30°.

The specimen rotation with 6 min^{-1} frequency leads us to consider that the stress state in Au thin films has rotational symmetry i.e. $\sigma=\sigma_2=\sigma_1$, σ_3= 0.

There were achieved the (311) line profiles of the specimens denoted Au-1, Au-2 and Au-3 for the tilting angles: ψ = 0°, 10°, 15°, 20° and 30 °. The used values of the Young's modulus and Poisson's ratio for electrolytic Au were: E_{Au} = 80 GPa and ν_{Au} = 0,42, respectively.

6. Results and discussions

The calculated values of the residual stress values for Au obtained in 5 repetitive conditions are presented in Table I. There were choosen 5 repetitive tests taking into account that 5 is the maximum order of multiconvolution we achieved and the XRD practice e.g. the norm is 3 repeted tests, thus 5 is more than better.

Table 1

Data regarding the specimens Au-1, Au-2 and Au-3

specimen	2θo	σ(MPa)	σ(MPa)	σ(MPa)	σ(MPa)	σ(MPa)	$\overline{\sigma}$ (MPa)
	No. of trials	1	2	3	4	5	Average
Au-1	33,737	334,4	348,3	321,5	339,2	329,4	334.56
Au-2	33,749	372,74	381,5	384	370,6	378	377,36
Au-3	33,742	367,4	359,1	365,3	372,2	370,6	366,92

At a first glance the values obtained by a quite complicated calculation procedure are wide spread about their mean as is shown in Fig 5.

The stresses in Au-2 specimen are systematically bigger than those in Au-1 and Au-3 specimens. But, if one takes into consideration the MU of the results then the Au-3 and Au-2 could not be differentiated as it is shown in the followings.

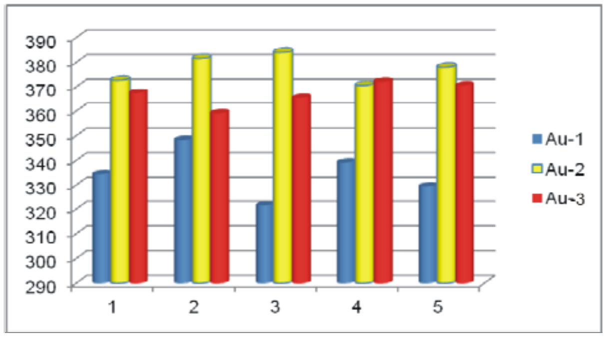

Fig. 5. Comparative outcome representation

In Table 2 are given the relevant results for this test i.e. the mean residual stress, the standard deviation (SD) of results, the SD of means, the maximum and minimum values of the estimated stresses for each specimen, the extended uncertainties having a confidence level of 95% estimated according to GUM approach (U_{GUM} (95%)) and the extended uncertainties estimated by multiconvolutional approach (U_{MCA}(95%)).

Table 2

U_{GUM} (95%)) and U_{MCA}(95%)

Specimen	Average stress value	SD	SD of mean	U_{GUM} (95%)	sigma max	sigma min	U_{MCA} (95%)
Au-1	335	10	5	9	348	322	6
Au-2	377	6	3	5	384	371	3
Au-3	367	5	2	5	372	359	3

As could be seen in Table 2, the GUM approach overestimates the U(95%) with 35 to 45% for each result. Using the specimen U_{GUM} (95%) of the mean one could not differentiate the specimens by the level of residual stress as could be seen in Fig. 6, while using the U_{MCA}(95%) the specimen could be arranged in the order of increasing residual stresses as: Au-1, Au-3, and Au-2 (fig.7)

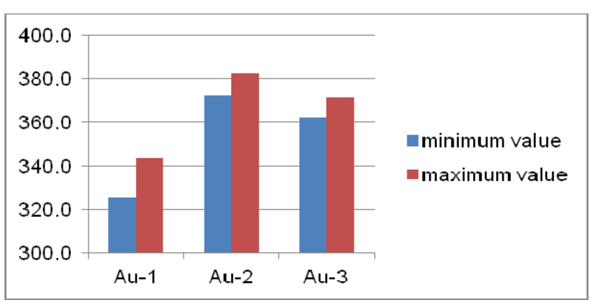

Fig. 6. The minimum values of residual stresses in the specimen (blue) and the maximum ones (red) estimated with U_{GUM} (95%)

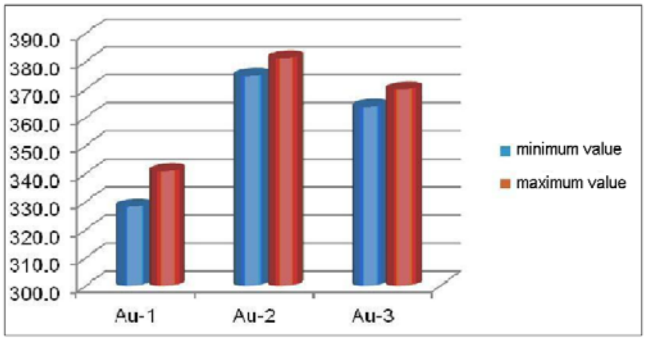

Fig. 7. The minimum values of residual stresses in the specimen (Series 1) and the maximum ones (Series 2) estimated with U_{MCA} (95%).

6. Conclusions

In order to underpin the advantage of our approach (MCA) comparing to the GUM approach [2], $U(95\%)$ of the results were estimated by both approaches.

The GUM approach overestimates the $U(95\%)$ with 35 to 45% for each result. As a consequence the GUM approach hinders the differentiation between Au-2 and Au-3 specimen by the stress level criterion while MCA really does it.

The MCA approach is more appropriate to the "$\sin^2(\psi)$" method for residual stress measurement because it does not imply any assumption about the pdf of the measurand while GUM approach assumes that the pdf of the measurand is of normal or Gaussian type.

We consider that there are other many tests that need a multiconvolutional approach of uncertainty estimation based on uniform probability density distributions.

R E F E R E N C E S

[1]. ***EN ISO/IEC 17025 - General requirements for the competence of testing and calibration laboratories,2005

[2]. *** SR Guide ISO/CEI 98-3- Guide to the Expression of Uncertainty in Measurement, 2010

[3]. *** ASTM E837 "Standard Test Method for Determining Residual Stresses by the Hole-Drilling Strain-Gage Method," 1994.

[4]. *I.Pencea*, Methods and Techniques of Instrumental Analysis of Materials in: Saban, R., editor, Treatise of Science and Engineering of Metallic Materials, AGIR Publisher, Bucharest, Romania, 2011.

[5]. *P.S.Prevéy, and P.W.Mason,* Practical Applications of Residual Stress Technology, ed. C. Ruud, Am. Soc. for Met., Materials Park, Ohio (1991).

[6]. *B. Eigenmann, V. Schulze and O. Vöhringer*, Proc.Int. Conf. on Residual Stress, ICRS IV, SEM, 1994, 598-607.

[7]. *M. B. Prime, R. J. Sebring, J. M. Edwards, D. J. Hughes, P. J. Webster*, Laser surface-contouring and spline data-smoothing for residual stress measurement, Experimental Mechanics, April 2004, Volume 44, Issue 2, pp 176-184.

[8]. *C. O. Ruud, P. S. DiMascio, J. J. Yavelak* , Comparison of three residual-stress measurement methods on a mild steel bar, Experimental Mechanics , December 1985, Volume 25, Issue 4, pp 338-343.

[9]. *Anton I. Lavrentyev, Paul A. Stucky, and William A. Veronesi*, Feasibility of ultrasonic and eddy current methods for measurement of residual stress in shot peened metals, AIP Conf. Proc. 509, pp. 1621-1628; doi:http://dx.doi.org/10.1063/1.1306227.

[10]. *Paul S. Prevéy*, A Method Of Determining The Elastic Properties Of Alloys In Selected Crystallographic Directions For X-Ray Diffraction Residual Stress Measurement, Advances In X-Ray Analysis, Vol. 20, Plenum Press, New York, Ny, (1977) Pp. 345-354.

[11]. *Hilley, M.E.*., ed, Residual Stress Measurement by X-Ray Diffraction, SAE J784a, pp. 12-15, NY: Society of Automotive Engineers, Inc. (1971).

[12]. *H. Zantopulos and C.F. Jatczak*, "Systematic Errors in X-Ray Diffractometer Stress Measurements Due to Specimen Geometry and Beam Divergence," Advances in X-Ray, Analysis, Vol. 14, pp. 260-376, 1971.

[13]. *Paul S. Prevéy*, Problems With Non-Destructive Surface X-Ray Diffraction Residual Stress Measurement, Www.Lambdatechs.com ▪, info@Lambdatechs.Com.

[14]. www.Lambdatechs.Com

[15]. www.rigaku.com

[16]. www.tec.com

[17]. *I. Pencea*, Chapter 6, “Multiconvolutional Approach to Treat the Main Probability Distribution Functions Used to Assess the Uncertainties of Metallurgical Tests” in the book “Metallurgy - Advances In Materials And Processes” ISBN 978-953-51-0736-1, Book edited by Dr. Yogiraj Pardhi, School of Metallurgy and Materials, University of Birmingham, UK; Published by InTech — Open Access Company, (http://www.intechopen.com), September 2012.

[18]. *Frank Killmann, F., von Collani, E.*, (2001) A Note on the Convolution of the Uniform and Related Distributions and Their Use in Quality Control, Heldermann Verlag Economic Quality Control, Vol 16 , No. 1, pp.17 – 41.

[19]. *Renyi, A.,* (1970) Probability Theory. North-Holland, Amsterdam, 458p.

[20]. ***Guide to the Expression of Uncertainty in Measurement, Edition, ISO, Geneva, 1995.

[21]. ***ISO 6508-1:2005, Metallic materials -- Rockwell hardness test -- Part 1: Test method (scales A, B, C, D, E, F, G, H, K, N, T)