Experimentální Analýza Napětí 2009
Experimental Stress Analysis

MICROSTRAINS AND X-RAY DIFFRACTION

Jan Drahokoupil[1,2], Marian Čerňanský[3] & Kamil Kolařík[4]

Abstract: Three additive kinds of residual stresses in a polycrystalline material are distinguished according to their corresponding length scales. An attention is paid to the microstrain – its effect on X-ray diffraction and a method to its estimation from a broadening of diffraction lines. Especially, the single line Voigt function is presented for the estimation of the microstrain and crystallite size from a single diffraction line.

1. Introduction

The XRD method is based on diffraction of X-rays on electrons. If the electron density has some periodical structure (e.g. crystallite), then also the sum of scattered X-rays bears the information on this structure. So that in XRD is observed the intensity of scattered X-rays on diffraction angle. The most often used Brag-Brentano geometry is shown (Figure 1). The basic positions and intensity of diffraction peaks give the information on mutual positions of atoms. The study of precise peaks positions, under several incident angles, enable determination of macroscopical stress. And lastly the breath of diffractions peaks is influenced by crystallite size and microstress. Since these two parameters of real structure have similar, but not the same, effect, have to be determinate together. For example high machined steel sample could have very different diffraction pattern (Figure 2); new peaks from new phases arise and peaks from original phase are broadened and shifted.

Most machine and structural materials have a polycrystalline structure that is formed by a large quantity of randomly oriented crystal grains. As a result of the different orientation of neighboring grains and anisotropy of elastic constants, yield strength and material strengthening, individual grains are deformed in elastic-plastic deformation differently. As a consequence of the uneven deformation in different grains, micro-stresses are generated which are in equilibrium in microscopically small volumes of material, comparable with the grain sizes.

[1] Ing. Jan Drahokoupil; Department of Metals, Institute of Physics of the ASCR, v. v. i., Na Slovance 2, 182 21 Praha 8, jandrahokoupil@seznam.cz
[2] Ing. Jan Drahokoupil; Department of Solid State Physics, FNSFE CTU in Prague, Trojanova 13, 120 00 Praha 2, jandrahokoupil@seznam.cz
[3] Ing., Marian Čerňanský, CSc.; Department of Metals, Institute of Physics of the ASCR, v. v. i., Na Slovance 2, 182 21 Praha 8, cernan@fzu.cz
[4] Ing. Kamil Kolařík; Department of Solid State Physics, FNSFE CTU in Prague, Trojanova 13, 120 00 Praha 2, kamil.kolarik@email.cz

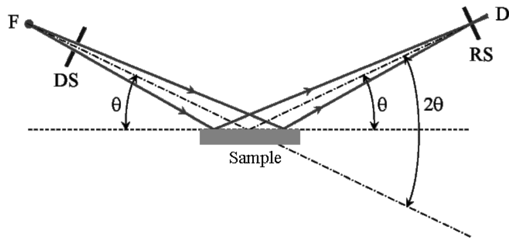

Figure 1: Basic settings of X-ray diffraction measurement (Bragg-Brentano focusing geometry). F- focus of the x-ray source, DS – divergence slits, RS – receiving slits, D – detector, θ – Bragg angle [1].

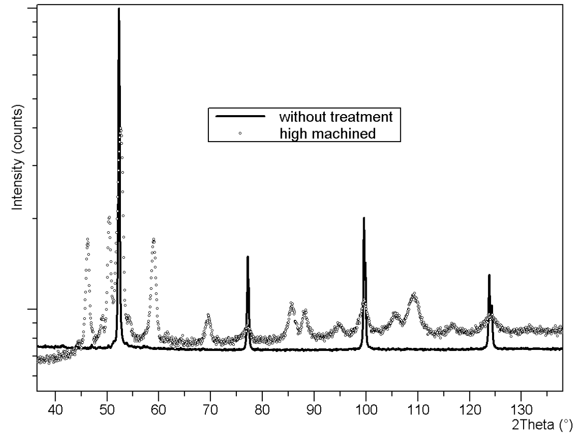

***Figure 2:** The comparison of diffraction pattern standard sample without treatment a high machined steel sample [2].*

2. Theoretical background

2.1. Strain and Stress

The interplanar distance d_{hkl} could be computed by Bragg's law (1) from diffraction angle 2θ which is measured. Differentiation of Bragg's law leads to relation between strain of diffraction planes e_{hkl} and diffraction angle 2θ (2).

$$2d_{hkl}\sin\theta = n\lambda \tag{1}$$

$$e = \frac{d - d_0}{d_0} = -\Delta\theta\cot\theta_0, \tag{2}$$

where index 0 denote the non-deformed interplanar distance. So it must be emphasize that by x-ray diffraction only strains are measured! The elastic constants have to be used to determination of stress. Many materials have anisotropic monocrystalline elastic constant. For example Young's modulus of monocrystal of Iron in crystallographic direction (*111*) E^{111} = 273 [GPa] and in crystallographic direction (*100*) E^{100} = 125 [GPa], [3]. X-ray diffraction, on contrary from other macroscopical methods of determination of residual stresses, distinguishes particular crystallographic directions. Moreover the monocrystaline grains in polycrystalic material are influence each other. So some special grain-interaction models should by used [4].

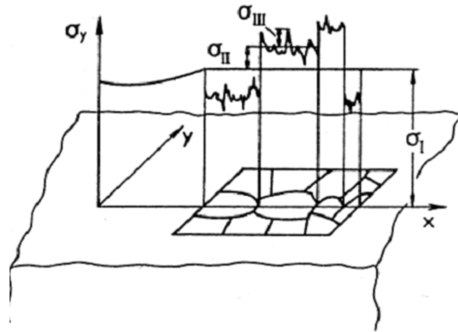

Figure 3: *The divisions of stresses [5].*

In the absence of external loads, stresses present in the sample are called residual stresses. Three additive kinds of residual stresses in a polycrystalline material are distinguished according to their corresponding length scales, (Figure 3).

where:
σ - local stress,
σ_I - the average of the residual stresses over many grains,
σ_{II} - the difference between the average of the residual stresses over a particular grains and σ_I,
σ_{III} - the deviation of a local stress σ in a particular grain from the average stresses in the grain.

2.2. Broadening of diffraction peaks due to microstrains

The impact of microstrains on X-ray diffraction profile is very good known [6]. To show this clearly a simple vision is presented. For simplicity only three peaks from three different grains under different stress are taken into account (in reality there are thousands of grains); each peak has the width that corresponds to instrumental broadening (others sample affect are for simplicity neglected). The "sum peak" denotes plain summation of intensities from the three particular grains. It is clearly visible that the presence of non-oriented microscopic stresses causes diffraction profile broadening (Figure 4).

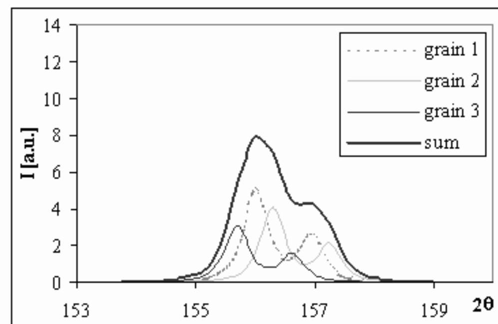

Figure 4: *The effect of microstrees.*

2.3. Determination of microstrains

It has been shown that microstrains cause broadening of diffraction peaks. The width of a diffraction peak is most often described by one of following two breadth parameters: integral breadth β or full width at half maximum *FWHM.* Both the peak parameters could be related to $\Delta\theta$ from equation (1) and then it would be transformed into [6]

$$e = \frac{1}{4}\beta \cot\theta_0 \tag{3}$$

Therefore, the microstrains can be determined from the knowledge of breadth β and position of diffraction angle θ_0. Nevertheless, the situation is not so simple in a real material, because additional sources of diffraction profile broadening exist there. Small crystallite size is another frequently occurring reason for broadening. Several methods are used to separate the influence of microstrains and crystallite size. The majority of them is based on a dependence of broadening on diffraction angle and uses several diffraction lines [7]. Single line Voigt function method, witch is discussed thereinafter, analyze the shape of diffraction peaks and is able to separate the broadening from crystallite size and from microstrains using one line only [8].

3. Single line Voigt function method

This method is based on the assumptions that the particle size leads to the Cauchy (Lorentz) profile and the microstrain is connected with the Gauss profile. The additional assumption is that both these factors are composed independently. Mathematically it means that the physical diffraction profile is a convolution of Cauchy and Gauss profiles, which is the Voigt function. The most important quantity is the shape factor φ of a diffraction line, which is defined as the ratio of the full width 2w at the half of the maximum (FWHM) to the integral breadth of the line β, i.e. $\varphi = 2w/\beta$.

The Cauchyian and the Gaussian part of the integral width of the Voigt function is given by the following relations

$$\beta_C = \beta\,(2.0207 - 0.4803\,\varphi - 1.7756\,\varphi^2) \qquad (4)$$

$$\beta_G = \beta\,(0.6420 + 1.4187\,(\varphi - \varphi_C)^{1/2} - 2.2043\,\varphi + 1.8706\,\varphi^2), \qquad (5)$$

where $\varphi_C = 2/\pi = 0.6366$ is the shape factor of the Cauchy function (the shape factor of the Gauss function is $\varphi_G = 2\,((\ln 2)/\pi)^{1/2} = 0.9394$). The maximal error of the relations (3) and (4) is approximately 1 %.

Relations (3) and (4) are used to decompose the integral width of a measured diffraction profile h on the Cauchyian part β_C^h and Gaussian part β_G^h. The same procedures are used for the instrumental profile g, which is obtained from a measurement on a convenient standard, without a diffraction (physical) broadening. The Cauchyian β_C^g and Gaussian β_G^g parts of the instrumental profile are estimated also from the relations (3) and (4).

The Cauchyian β_C^f and the Gaussian β_G^f parts of the integral width of the physical profile f are determined from relations

$$\beta_C^f = \beta_C^h - \beta_C^g \qquad (6)$$

$$(\beta_G^f)^2 = (\beta_G^h)^2 - (\beta_G^g)^2 \qquad (7)$$

The crystallite size D and the microstrain ε are given by the relations

$$D = \lambda\,/\,(\beta_C^f \cos\theta) \qquad (8)$$

$$\varepsilon = \beta_G^f\,/\,(4\,\mathrm{tg}\,\theta), \qquad (9)$$

where λ is the wave length and θ is Bragg angle.

4. Conclusions

The X-ray diffraction is a useful experimental technique which enables to estimate the microscopic stresses and many other characteristics of material. For surface values (several μm) is this method non-destructive, without any preparation of sample surfaces. The effect of broadening of diffraction peaks due to microstrains is illustrated by Figure 4. The Single line Voigt function method enable determination of microstrain using only one diffraction line. This leads to possibility to use the same line to determine both macroscopical and microscopical strain and thereby avoid the problems with elastic anisotropy. This method also determine the grain size.

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