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STRUCTURE IRREGULARITIES DETECTED BY X-RAY DIFFRACTION EFFECTS

EFEKTY DYFRAKCYJNE A NIEREGULARNOŚCI STRUKTURY

Polycrystalline materials are solids which properties depend on such quantities as chemical and phase composition, crystallographic texture, residual stresses, grain size, etc. Applied technological processes lead usually to change the values of the parameters describing the quantities as well as modify the structure characteristics in macro- and micro-scale. In the result, both the global and local irregularities of material structure appear. When the irregularities become significant, numerous material properties reveal differences from point to point in a prepared constructing element. Spatial distribution of the irregularities can show a continuous character (gradient of the properties) or a non-continuous one, like in a layered structure. Moreover, the mentioned structure heterogeneity can be an intended effect like in a functionally graded materials or a quite non-favorable result of technological process (e.g. non controlled grain growth in the heat affected zone of welded elements). Among the most provocative challenge for the researchers are the structure inhomogeneities appeared under exploitation conditions (e.g. fatigue wear of the near-surface areas). In spite of the origin of the above structure irregularities, great research problem is recognizing its spatial distribution in the material. One of the most effective and non-destructive tool in this range is the X-ray diffraction technique assisted by appropriate experimental method and data processing procedures.

The work presents the changes of the diffraction peak parameters of structure irregularities of a welded constructing element analyzed by the X-ray technique applied to investigation.

Materiały polikrystaliczne są ciałami stałymi, których własności zależą od takich własności jak skład chemiczny i fazowy, tekstura krystalograficzna, naprężenia własne, rozmiar ziarna itp. Zastosowane procesy technologiczne prowadzą zwykle do zmiany wartości parametrów opisujących te własności, jak również modyfikują strukturę materiału w makro- i mikro-skali. W rezultacie w materiale pojawiają się globalne i lokalne niejednorodności. Kiedy nieregularności stają się znaczące, wiele cech materiału zmienia się od punku do

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punktu w tak przygotowanym elemencie konstrukcyjnym. Przestrzenny rozkład tych niejednorodności może mieć charakter ciągły (gradient własności) lub nieciągły, jak w strukturze warstwowej. Co więcej ta niejednorodność może być zamierzonym efektem, czego przykładem są funkcjonalne materiały gradientowe, lub niechcianym rezultatem technologicznego procesu (np. niekontrolowany wzrost ziaren w strefie wpływu ciepła w elementach spawanych). Wyzwaniem dla badaczy są niejednorodności powstałe w warunkach eksploatacyjnych (np. zmęczenie materiału w warstwach powierzchniowych). Niezależnie od pochodzenia tych nieregularności, problemem badawczym jest poznanie przestrzennego rozkładu tych efektów w materiale. Jedną z najbardziej efektywnych i nieniszczących metod jest dyfrakcja rentgenowska, przy wsparciu odpowiednich technik eksperymentalnych i przetwarzania uzyskanych danych.

Przedstawiona praca poświęcona jest zmianom w parametrach pików dyfrakcyjnych uzyskanych przy użyciu dyfrakcji rentgenowskiej w konstrukcyjnym elemencie spawanym.

1. Introduction

Measurements of materials properties on relatively large areas using X-ray diffraction technique may become a very effective tool for detecting various types of spatial irregularities over the investigated sample. One of the most important advantages of using X-ray beam is relatively easy sample preparation (in many cases no particular preparation is needed) as well as a large sample area (in presented case a sample stage x-y-z is capable of scanning 80×80mm sample area). Because of the method being non-destructive, investigated sample can be still applied as a constructing element.

Such type of information can be obtained from the material using typical X-ray diffractometer with sample stage capable of moving the sample in a chosen measurement grid and a suitable primary beam optics producing narrow, well defined beam. Peak shape, intensity and position changes gives information about change of properties. Usage of modern detector (area or linear one) is favourable, to reduce total measurement time. One can perform two types of measurement routine of the sample:

- peak parameters topography (PPT)
- pole figure topography (PPF)

Peak parameters topography consist in registration of chosen line profile(s) with narrow primary beam in respect to sample position on the stage in the given measurement grid, as it is shown on Fig. 1. From the line profiles, after appropriate processing one can obtain many useful information on material characteristics in a given "point". By point authors means small sample area where the diffraction effect occurred. Then the series of topographic maps presenting various material characteristics can be constructed.

The pole figure topography (PFT) differs when compared to the PPT in fact that instead of the line profile one have to obtain pole figure (or a part of it). It is much more time consuming (because measurement time for typical pole figure which consists of 10^3 profiles is larger by the same order of magnitude). However the pole figure gives more detailed information about properties of surface layer when compared to pure, one peak profile.

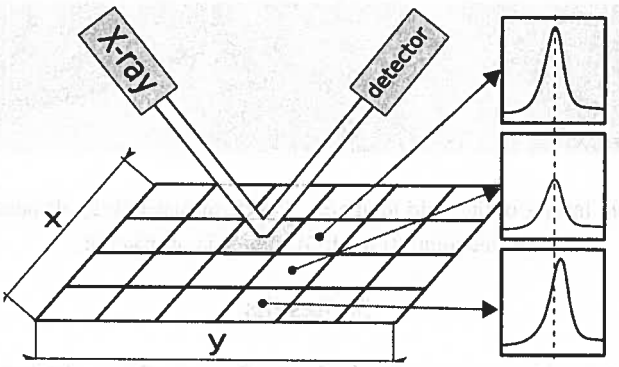

Fig. 1. Obtaining X-ray line profiles map by scanning over given grid on the sample surface

2. Measurements

Diffraction experiments were performed in Institute of Materials Physics, University of Vienna using Bruker AXS diffractometer D-8 Discover with GADDS area detector and x-y-z sample stage. Investigated sample was a cross section of welded steel sheets. View form the video camera mounted on diffractometer stand is presented in Fig. 2. Some areas of the sample were also investigated using optical microscope, to analyse the material microstructure (Fig. 3).

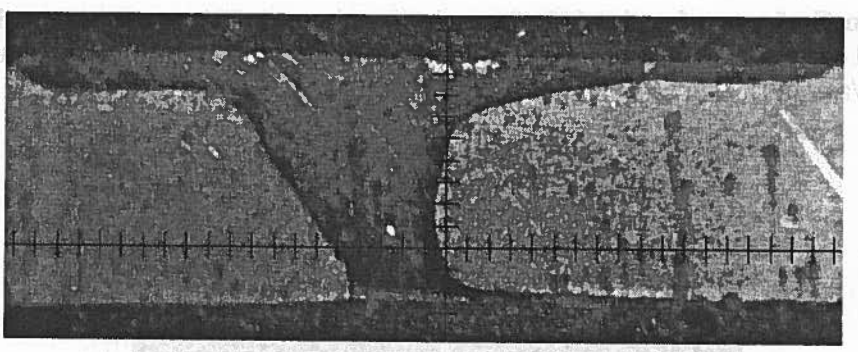

Fig. 2. 2D view of the investigated sample surface from the video camera mounted on experimental stand

Examined sample was measured on the grid with $0.2mm$ step size, resulting in the grid with $13 * 52 = 676$ points, covering $2.6 * 10.4 = 27.04mm^2$. Two peak profiles — 110 ferrite and 111 austenite were collected in every point of the grid. Obtained line profiles were automatically fitted by theoretical profiles [1]. From such created data maps of the selected peak profile parameters were constructed and visualised [2].

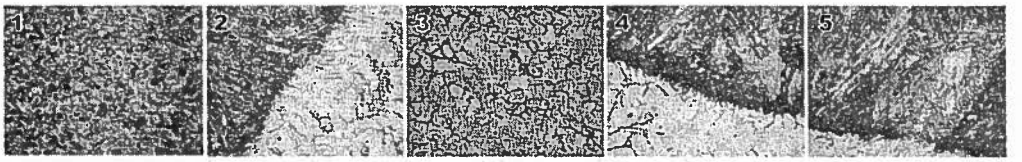

Fig. 3. Microstructure images of the weld joint area. 1) side of material; 2), 4) border between material and the joint; 3) joint; 5) right side of material

3. Results

Experimental results are presented in form of maps of the selected peak parameters. Presented on Fig. 4 is ratio of maximal intensity of profile from ferrite to intensity from austenite. The ratio reflect distribution of volume fraction of both phases.

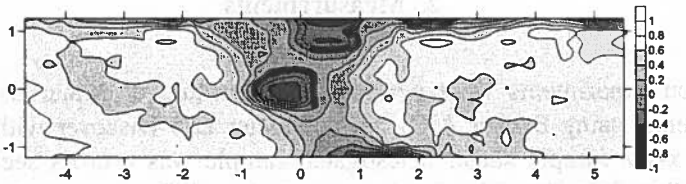

Fig. 4. Maximal intensity of diffraction peak for ferrite and austenite in weld joint sample. Value of +1 on the vertical scale means 100% ferrite and 0% austenite. Value of −1 have the opposite meanin

On Fig.5 map of peaks shift (2θ angle) is presented. This peak shift can be connected with the 1st order residual stresses in the sample as well as admixtures in ferrite/austenite.

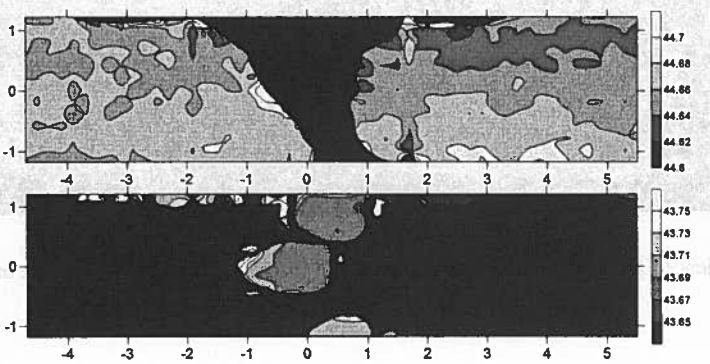

Fig. 5. Topographic maps of peak shift

On Fig. 6 map of FWHM of peaks is presented. Width of the diffraction peak depends on size of grains (coherent domains) and residual stress of 2nd order.

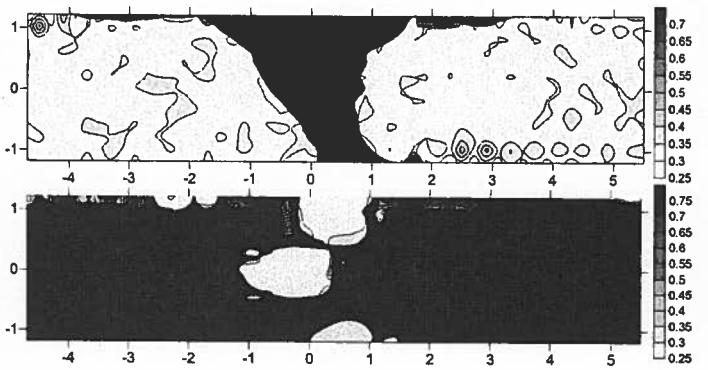

Fig. 6. Topographic maps of peak FWHM

4. Conclusions

Among many diffraction effects one can register form sample using x-y-z-ϕ-χ stage most interesting for further investigations are pole figures and line profiles. Those measurement allow to characterise material's surface by means of:

- Inhomogeneity of texture
- Configuration of stresses field,
- Phase distribution over sample area,
- Distribution of thickness of surface layer,
- Localisation of strong plastic deformation areas in materials.

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