A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
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PLANAR IRREGULARITIES OF TEXTURE AND STRESS FIELD DETECTED BY X RAY DIFFRACTION TECHNIQUE

PLANARNA NIEJEDNORODNOŚĆ TEKSTURY I POLA NAPRĘŻEŃ IDENTYFIKOWANA TECHNIKĄ DYFRAKCJI RENTGENOWSKIEJ

Regardless of the origin of structure irregularities of materials, recognizing its spatial distribution in a sample or constructing elements is a great research problem. One of the most effective and non-destructive tools in this range is the X-ray diffraction technique assisted by appropriate experimental method and data processing. The work presents the results of investigations of planar distribution of crystallographic texture and stress irregularities manifested by changes of diffraction effects registered by X-ray technique. As an example, the introduced method is tested on titanium rod after severe plastic deformation process.
Keywords: titanium, X-ray diffraction, crystallographic texture, residual stresses

Identyfikacja przestrzennego rozkładu niejednorodności struktury materiału, bez względu na przyczyny ich powstania, stanowi istotny problem badawczy. Jednym z najbardziej efektywnych i nieniszczących narzędzi badawczych w tym zakresie jest technika dyfrakcji rentgenowskiej. W niniejszej pracy zaprezentowano wyniki badań rozkładu planarnego tekstury krystalograficznej i naprężeń własnych przejawiających się w zmianie charakterystyki odbić dyfrakcyjnych. Wprowadzona metoda topografii tekstury i naprężeń własnych została przetestowana na prętach tytanu poddanych silnemu odkształceniu plastycznemu.

1. Introduction

Properties of solid polycrystalline materials depend on such quantities as chemical and phase composition, crystallographic texture, residual stresses, grain size, etc. Applied technological processes usually change the values of the parameters described by those quantities as well as modify the structure characteristics in macro- and micro-scale. As the result, both the global and local irregularities of material structure appear. When the irregularities become significant, numerous material properties reveal differences from place to place in the prepared constructing component. Spatial distribution of the irregularities can show a continuous character (gradient of the properties) or a no continuous one, like in a layered structure. Moreover, the mentioned structure inhomogeneities can be an intended effect like in a functionally graded materials or a non favourable result of technological process (e.g. non controlled grain growth in the heat affected zone of the 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).

Recognizing the distribution of crystallographic texture on surface of constructing elements is desirable from technological as well as research points of view. So far, good known and sophisticated electro-diffraction techniques (e.g., EBSD-OIM [1]) delivers needed information about the space distribution of crystallographic orientation. However, the examined volume of material is too small regarding the observation scale of investigated effects, e.g. structure of composite materials, junctions of construction elements, coarse grained materials, and deformed single-crystals.

Idea of getting the large-area (a few cm^2) distribution of texture changes or residual stresses has been based on relatively easy to achieve X-ray diffraction technique. It consists in determined registration mode of the diffraction effects, computer controlling of measurement and data processing [2-4]. The work presents as an example results of the X-ray topography of texture and

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residual stresses existed in a longitudinal section of Ti ingot subdued to severely plastic deformation by means of the ECAP processing.

2. Material and Experimental

Commercial purity Ti (99.95%) was used as investigated material. The size of the billet subjected to the ECAP processing was 60·8·8 mm^3. Temperature of the ECAP processing of the billet was equal to 450°C and deformation rate was 6 mm/s. The angle of channels intersection during ECAP processing was 90° [5]. Sample was prepared from material subdued to ECAP processing by Bc route interrupted in the middle of second pass (see Fig. 1). The Ti billet was removed and then cut with respect to vertical plane. Scheme of this process is shown in Fig. 2. X-ray investigations were made in the longitudinal section of the geometrical centre of the Ti billet. Incomplete pole figures were measured in selected areas neighbouring to the knee of the ECAP tool.

Topographic measurements were performed using the filtered CuK radiation by means of Bruker D-8 Discover diffractometer, equipped with the open Euler cradle (Fig. 3). X-ray tube radiation was focused using polycapillar optics ensuring a near-parallel incident beam. The beam size was approx. 1 mm in diameter. Two pole figures (0002) and (10-11) of Ti in chosen 31 areas on sample surface (as shown in Figs. 4-6) were collected. Experimental data were analyzed using software developed at the Institute of Metallurgy and Materials Sciences of the Polish Academy of Sciences in Krakow [3-5]. Residual stresses analysed in the measured areas of the sample were calculated based on a recent approach in the field [6].

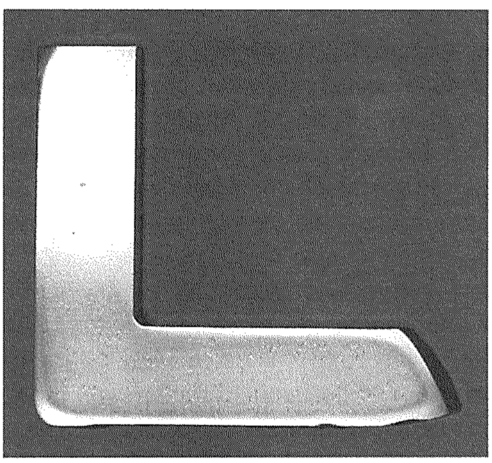

Fig. 1. Ti sample prepared for diffraction experiment

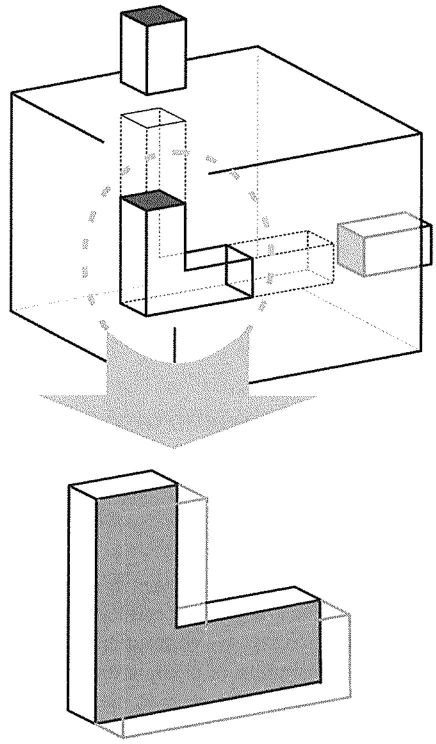

Fig. 2. Sketch of ECAP processing tool. Material from which sample was prepared was removed in the middle of the second pass (Bc route). Then it was cut in respect to the plane shown on bottom part of the picture

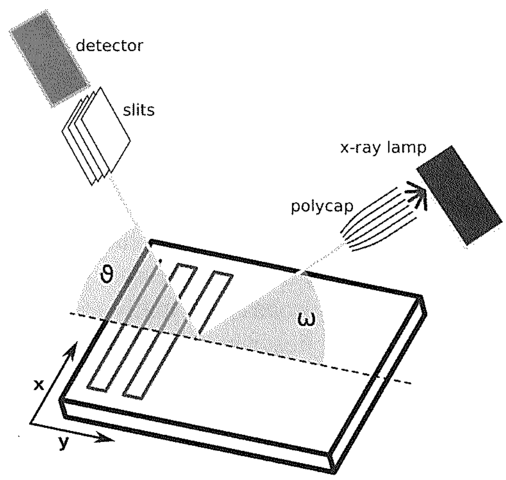

Fig. 3. Scheme of the X-ray topography experiment with trace of measurement path on surface of examined sample

3. Results and discussion

Orientation Distribution Function (ODF) of the sample for each of the 31 measured sample areas was calculated. Then the complete pole figures as well as the inverse pole figures on direction normal to the sample surface were created. Chosen results in form of a topographic distributions of texture referred to investigated area of the sample are presented in Figs. 4 and 5. Maximal values of the ODF are located in a post-bisectional zone of the ECAP tool. In the bisection region of the angular channel the most intensive shearing take place during the deformation process. For that reason also the most intensive changes of texture, manifested also in the ODF values are expected. On the other hand, formation of the texture observed in the longitudinal section the billet reveals differences in it formation dependent on deformation rate in the investigated area of sample. Such conclusion can be withdrawn based on a planar distribution of maximal values of inverse pole figure of the direction normal to sample surface presented in Fig. 5. More detailed analysis of texture components (not presented here) indicates differences in mechanisms of deformation activated in the individual sub-areas of investigated bended zone of the sample.

Registered pole figures include besides of a real space distribution of crystallographic poles also information on residual stresses which can be extracted from the experimental data [7]. Results of performed calculations, regarding isotropic elastic constants for Ti are presented in Fig. 6.

Identified stresses are compressive nature in the whole investigated area of the sample. It can be noticed that planar distributions of the mutually perpendicular stress components σ_{11} and σ_{22} are essentially different and a strong anisotropy of the stress field is evident. The highest values (module values) of the σ_{11} component appear in the left and bottom side of the bended area of sample. The σ_{22} component indicates maximal values in the post bisection region of investigated sample area. At the same time, along the bisection zone, the both components reach a relative middle values what can suggest a relaxation processes occurred into this volume of material.

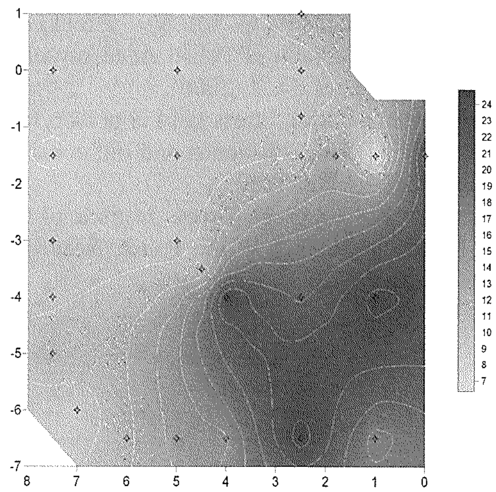

Fig. 4. Planar distribution of maximal values of the ODF calculated from incomplete pole figures

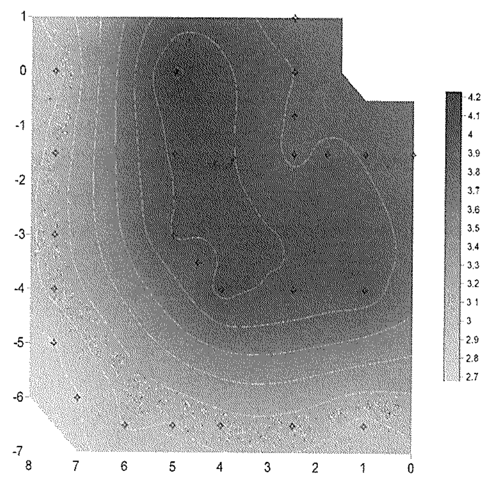

Fig. 5. Planar distribution of maximal values of inverse pole figure of the direction normal to sample surface calculated from the ODF

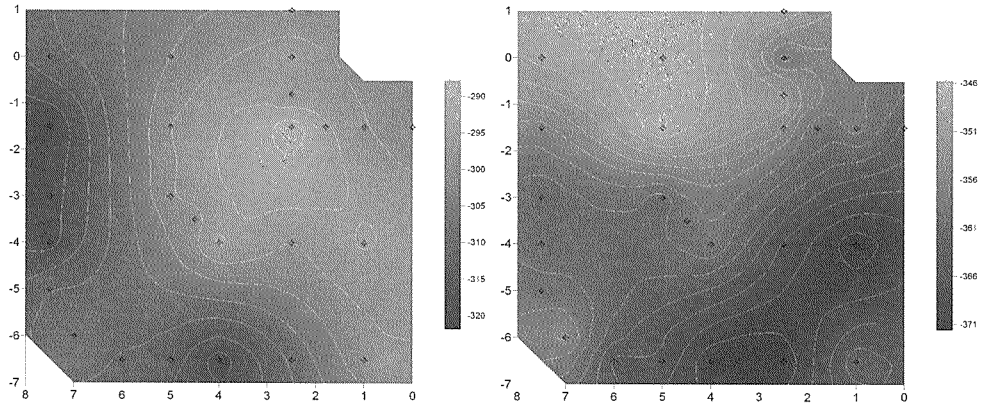

Fig. 6. Planar distribution of σ_{11} (parallel to vertical direction) and σ_{22} (in horizontal direction) stress components (values in MPa) in investigated sample area

4. Conclusions

Analyzed fragment of the processed Ti ingot (with hexagonal lattice symmetry) belongs to the most crucial sample area from viewpoint of deformation mechanism of severely plastically deformed material by the ECAP method. Most intensive shearing, fragmentation and rotations of grains take place in processed material in the bended part of the tool. As a result, under the local stress configuration a specific texture of Ti is formed. Analysis of the diffraction effects in the chosen areas of the sample surface enable to identify the topography of texture and residual stresses generated during the ECAP and allow to form the following conclusions:

- Planar distribution of the identified residual stresses and crystallographic texture in the middle plane during ECAP process of Ti ingot is strongly inhomogeneous.
- Character of the above irregularities reflects mechanism of deformation of Ti in the most crucial zone of the ECAP tool.
- Maximal intensity of the inverse pole figures of the normal direction is close to the (10-11) crystallographic direction which correspond to the twinning plane of deformed Ti. Additionally, localization of the maxima on the sample contour indicate zone of the possible sliping/twinning in the processed material.
- Diagonal components σ_{11} and σ_{22} of the residual stresses reflect nature of acting forces during the ECAP processing of the Ti ingot.

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