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Extinction Phenomenon in X-Ray Diffraction Technique for Texture Analysis

Fenómeno de extinción en la técnica de difracción de rayos X para el análisis de textura

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Abstract

A method to correct pole densities (PD) for primary and secondary extinction applied for maxima of pole figures (PF) measured by X-ray diffraction, was extended to correct the whole 111 and 200 PFs for nickel samples after 75% cold rolling and subsequent annealing at 600°C during 30 minutes. The PDs were corrected, and parameters of primary and secondary extinction were calculated using the PDs obtained in PFs measured for the first order reflections with two wavelengths (Cu Kα and Co Kα - radiations) and for the second order reflections with Cu Kα – radiation. Three orientation distribution functions (ODF) were calculated, namely: the first one from 111, 200 and 220 PFs; the second one from 222 and 400 PFs (the second order reflections) and 220 PF (440 reflection is absent for the radiations used); the third one from corrected 111 and 200 PFs and not corrected 220 PF (for lack of the second order reflection). Essential differences between the obtained ODFs indicate the necessity to take into account the extinction phenomenon in analysis of textured materials. The obtained parameters of extinction were used for the evaluation of microstructure details of textured nickel depending on grains orientation that is not easily obtained by conventional metallographic methods.

Keywords:

- X-ray diffraction
- texture
- primary extinction
- secondary extinction
- microstructure

Resumen

Un método para corregir densidades de polos (DP) por extinción primaria y secundaria en máximos de figuras de polos (FP) medidos por difracción de rayos X, se extendió a todos los datos de las FP 111 y 200 de muestras de níquel con 75% laminación en frío y recocido posterior de 600°C durante 30 minutos. Las DP fueron corregidas, y los parámetros de extinción primaria y secundaria fueron calculados utilizando las DP medidas en FP para las reflexiones de primer orden con dos radiaciones (Cu Kα y Co Kα) y para las reflexiones de segundo orden con radiación de Cu Kα. Se calcularon tres funciones de distribución de orientación (FDO): la primera a partir de FP para reflexiones 111, 200 y 220, la segunda a partir de las FP 222, 400 (los segundos ordenes) y 220 (en ausencia de segundo orden) y la tercera con las FP para las reflexiones 111 y 200 corregidas por extinción y la FP 220 no corregida (en ausencia de segundo orden). Las diferencias esenciales entre las FDO obtenidas, indican la necesidad de tomar en cuenta el fenómeno de extinción en el análisis de materiales con textura. Los parámetros de extinción obtenidos fueron utilizados para evaluar los datos de la microestructura para níquel en función de la orientación de los granos, que no es fácil obtener por métodos convencionales metalográficos.

Descriptores:

- *difracción de rayos X*
- *textura*
- *extinción primaria*
- *extinción secundaria*
- *microestructura*

Introduction

In poly crystal line materials, grains are rarely randomly distributed from the point of view of crystallographic orientation. The preferred crystallographic orientation, or texture, arises during the manufacturing processes owing to anisotropic properties of individual grains and in turn, it affects different material properties (Bunge, 1987) and the correct evaluation of texture is important for materials research. The common method for quantitative texture analysis by X-rays diffraction (XRD) from a crystalline medium is based on the measurement of pole figures (PF) (Bunge, 1996; Kocks *et al*., 1998; Randle *et al*., 2000) that are characterized by pole densities (PD) obtained from the measured X-ray reflected intensities. The data of two-dimensional PFs are used for calculating a three-dimensional representation of the orientation density in terms of the orientation distribution function (ODF).

Since the initial information to obtain a PF is the data of reflected intensities, the fact that in PF measurements the extinction phenomenon can be present should be taken in to account. Indeed, it has been shown that extinction is present in PF measurements, and specially in those of low index reflections (Kryshtab *et al*., 2004; Tomov, 2011; Palacios-Gómez *et al*., 2010) with X-ray as well as with neutron diffraction, and a brief proof of the presence of this phenomenon can be easily done by comparing the PDs from PFs for higher order reflections with those for the first order reflections; here only PDs in maxima of PFs have been analyzed. If this phenomenon is present, it cannot be avoided and it reduces PDs in PFs for the first order reflection as compared to PDs in PFs for higher order reflections.

On the other hand, neither PFs nor ODFs contain information of microstructure details such as grain size and lattice perfection of grains. The conventional method for evaluation of microstructure in polycrystalline materials using XRD is based on the diffraction peak broadening (Warren, 1969; Langford, 1978) and kinematics cattering theory (Krivoglaz, 1996). It is impossible to use this method in the case of real crystals with low dislocation density, where full-width at half maximum of the peak reaches the instrumental breadth (Authier *et al*., 1980). In such crystals the X-ray dynamic scattering processes come into being and lead to the extinction phenomenon. The characteristics of extinction have been introduced into the kinematic theory in the form of two different phenomena (Darwin, 1922; Zachariasen, 1963) as the *primary extinction*, which depends on the size of coherently diffracting crystallites, and the *secondary extinction*, which depends on disorientation of crystallites. Therefore, the characteristics of extinction relate to the crystal microstructural feature and can be used for its evaluation.

An original technique for correction of the PD in the maximum of a PF and for the evaluation of grain microstructure using the characteristics of the primary and secondary extinctions was proposed by (Kryshtab *et al*., 2004), and was applied to an aluminum sample. Whereas the problem of the separation of contributions of PD and extinction phenomenon in XRD measurements of textured materials, when primary and secondary extinctions are present simultaneously, cannot be solved exactly, some reasonable assumptions were pro-