MS.32.5

Acta Cryst. (2011) A**67,** C83

Crystalline texture in Zr based alloys tubes

Constanza P. Buioli, Abraham D. Banchik, Pablo Vizcaíno, *Laboratorio de Materiales y Fabricación de Aleaciones Especiales (LMFAE) , Centro Atómico Ezeiza, Comisión Nacional de Energía Atómica (CNEA), Ciudad de Buenos Aires-Buenos Aires (Argentina).* E-mail: cpbuioli@cae.cnea.gov.ar

Pressure tubes are the most important Zr-base components in the core of CANDU nuclear power reactors. The most important mechanisms defining the service life of pressure tubes are: irradiation-enhanced coming with deformation, and the delayed hydride cracking. Both processes are determined by a few metallurgical parameters which must be exhaustively controlled: microstructure, dislocation density and texture [1], [2]. Argentina has one CANDU power plant in operation since 1982 (CNE), which is now close to ending its service life. A refurbishment project is underway which will include the replacement of existing fuel channels, composed by Zr-2.5%Nb pressure tubes and Zircaloy2/4 calandria tubes. CNEA is developing a new manufacturing route for Zr-2.5%Nb pressure tubes, using a cold rolling pilger type machine instead of the cold drawing process currently performed in Canada. The new process will be performed at the Planta Piloto de fabricación de aleaciones especiales (PPFAE,CNEA).

The extrusion stage produces a strong texture (crystalline preferential orientation) in the material. In this framework, in our laboratory (LMFAE), we calculate the texture factors [3], [4] from x-ray diffraction diagrams obtained from a Bruker D8 FOCUS diffractometer. A qualitative study of the texture was performed using the rocking curve method [5] (figure 1). The collected data was used to build a qualitative direct pole figure. The quantitative study was made through the calculation of Kearns factors (table 1), measuring in the θ/2θ standard way (Bragg-Brentano geometry). The results obtained from the rocking curves were quite satisfactory and they are compatible with the expected texture for the (0002) pole. This pole is essentially oriented in the transverse direction (90%), with small contributions in the radial (8%) and axial directions (2%). The Kearns factors are also in agreement with the these fractions, fulfilling the specified values ($0.03 \leq f_{axial} \leq 0.09$, $0.27 \leq f_{radial} \leq 0.39$, $0.55 \leq f_{transversal} \leq 0.67$, and $0.95 \leq \sum f \leq 1.05$ [6]) within a dispersion of ±0.02.

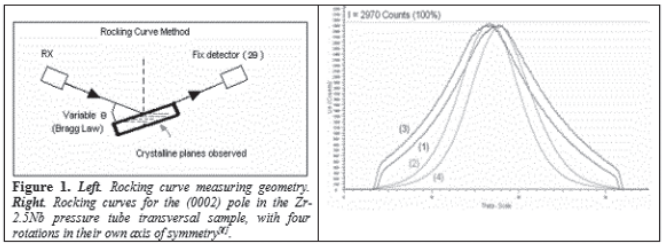


Figure 1. ***Left.*** *Rocking curve measuring geometry.* ***Right.*** *Rocking curves for the (0002) pole in the Zr-2.5Nb pressure tube transversal sample, with four rotations in their own axis of symmetry*[4].

Table 1. *Average Kearns factors of the Zr-2.5%Nb pressure tube.*

Axial factor	Radial factor	Transversal factor	Total factor
0,045	0,34	0,54	0,93

[1] J.R. Santisteban, M.A. Vicente-Alvarez, P. Vizcaino, A.D. Banchik, J.D. Almer, *Acta Materialia* **2010,** *58,* 20, 6609-6618. [2] K.B. Colas, A.T. Motta, J.D. Almer, M.R. Daymond, M. Kerr, D.A. Banchik, P. Vizcaino, J.R. Santisteban, *Acta Materialia* **2010,** *58,* 20, 6575-6583. [3] L.J. Galeta, C.P. Buioli, A.D. Banchik, P. Vizcaíno, *V Reunión de la Asociación Argentina de Cristalografía,* **2009**. [4] J.J. Kearns, *WAPD TM-472, Westinghouse Electric Corp.,* Pittsburgh, PA, **1965**. [5] C.P. Buioli, A.D. Banchik, P. Vizcaíno. *XI Congreso Iberoamericano de Metalurgia y Materiales y X Congreso CONAMET/SAM,* **2010.** [6] AECL, Qualification Requirements, Pressure Tube Manufacturer Qualification, CANDU COMMON, 00-31110-TD-001 Revision 0, **2006.**

Keywords: texture, zirconium, alloy.

MS.33.1

Acta Cryst. (2011) A**67,** C83

Low-voltage electron diffractive imaging

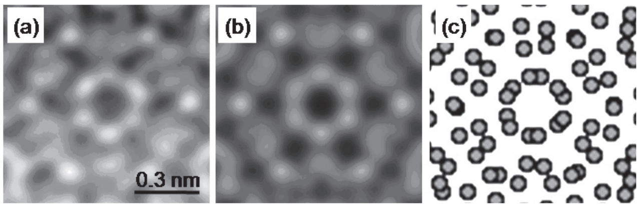
Osamu Kamimura,[a,b] Takashi Dobashi,[a] Yosuke Maehara,[b] Kazutoshi Gohara,[b] [a]*Central Research Laboratory, Hitachi Ltd., Tokyo (Japan).* [b]*Division of Applied Physics, Graduate School of Engineering, Hokkaido University, Sapporo (Japan).* E-mail: osamu.kamimura.ae@hitachi.com

Atomic-scale analysis demands of three-dimensional (3D) materials composed of light elements (e.g., carbon, lithium, boron) has been increased accompanied to spreading of the materials applications. For the materials, one of the most serious issues is the radiation-sensitive nature of the elements. Low-voltage (low-energy) electron diffractive imaging has the potential possibility to clarify the atomic-scale structure of 3D materials without causing knock-on damage to the specimen.

We have demonstrated low-voltage electron-diffractive imaging [1] by using a scanning electron microscope (SEM) based dedicated microscope [2] and a concomitant developed iteration algorithm [3, 4]. A 0.34-nm carbon wall spacing is resolved in multi-wall carbon nanotubes at an acceleration voltage of 10 kV [2]. Moreover, the atomic structure of single-wall carbon nanotube (SWCNT) with a resolution of 0.12 nm is obtained at 30 kV, and in the reconstructed pattern, the intensity difference between a single carbon and two overlapping ones is clearly distinguished [5]. An example of this is shown in the Figures below. Figure (a) shows the reconstructed pattern, Fig. (b) illustrates the simulated exit wave amplitude, and Fig. (c) is a model of the projected atomic structure of the SWCNT.

The use of a low-voltage electron beam is advantageous in the structural analysis of light-element materials, because lower voltage electrons strongly interact with atoms. The proposed SEM-based low-voltage electron diffractive imaging has the peculiar functions of SEM (e.g., ultra-low voltage function of voltage contrast) and TEM (atomic-resolution imaging). Our method will lead to a better understanding of the analysis results from the nanomaterials used in various fields and open up several possibilities in the developments of them.

We would like to thank Prof. Shinohara's group at Nagoya University for growing the SWCNTs and Prof. Shioya at the Muroran Institute of Technology for his invaluable contribution to the iterative algorithm. Part of this work was supported by Japan Science and Technology Agency (JST).



[1] O. Kamimura, K. Kawahara, T. Doi, T. Abe, K. Gohara, *Appl. Phys. Lett.* **2008**, *92*, 024106. [2] O. Kamimura, T. Dobashi, K. Kawahara, T. Abe, K. Gohara, *Ultramicroscopy* **2010,** *110*, 130. [3] K. Kawahara, K. Gohara, Y. Maehara, T. Dobashi, O. Kamimura, *Phys. Rev. B* **2010,** *81*, 081404(R). [4] H. Shioya, Y. Maehara, K. Gohara, *J. Opt. Soc. Am. A* **2010,** *27*, 1214. [5] O. Kamimura, Y. Maehara, T. Dobashi, K. Kobayashi, R. Kitaura, N. Shinohara, H. Shioya, K. Gohara, *Appl. Phys. Lett.* **2011,** in press.

Keywords: diffraction, electron microscope, phase retrieval