s8.m28.p14 **XRD Analysis of Tungsten Thin Films.** I. Djerdj[a], A. M. Tonejc[a], A. Tonejc[a] and N. Radic[b], [a]*University of Zagreb, Faculty of Science, Dept. of Physics, Bijenicka 32, P.O.Box 331, 10002 Zagreb, Croatia,* [b]*Ruder Boskovic Institute, Bijenièka c. 54, POBox 180, 10001 Zagreb, Croatia. E-mail: andelka@phy.hr*

Keywords: Tungsten thin film; X-ray diffraction; Phase composition

The properties of dc-magnetron sputtered thin tungsten films depend very strongly on deposition conditions. Depending on Ar gas pressure, substrate material, substrate temperature and deposition duration, there is a different relative amount of beta- and alpha-W phase deposit. In this work tungsten thin films prepared under different conditions of working-gas pressure and deposition temperature were investigated by means of X-ray diffraction (XRD) on a Philips powder diffractometer (PW 1820) and subsequently refined by the Rietveld method using the FULLPROF program [1]. The goal of this work was to investigate the dependence of microstructure and phase composition on deposition condition. Moreover, the microstructure, size and microstrain were extracted by different methods: Rietveld method, single-line method and Warren-Averbach method. The structures were refined in the space group of α-W, *Im-3m* and β-W, *Pm-3n*. In order to make size-strain analysis within the Rietveld method, the chosen diffraction profile function was the modified Thompson-Cox-Hastings pseudo-Voigt, with Lorentzian for size and Gaussian for microstrain. The scale factors, the background coefficients, the zero point of the detector, the lattice parameters, the Gaussian and Lorentzian halfwidth parameters were simultaneously refined. The quantitative phase composition was estimated from refined values of scale factors according to the procedure of Hill and Howard [2]. The single-line method was applied to selected well-defined non-overlapped diffraction maxima, assuming a Voigtian profile function. Last but not least, the determination of the Fourier size and microstrain coefficient using the so-called "double Voigt" method [3], which is equivalent to Warren-Averbach method, was used as an additional method for size-strain analysis. The basic requirement which must be fulfilled for the application of "double Voigt" method is the presence of two orders of reflection. This method is then applied to (200)-(400) reflections of β-W. It was found that at the deposition temperature of 293 K, on increasing the working-gas pressure, the amount of b-W phase increases. However, at low (77 K) and high (523 K) deposition temperature, on increasing the working-gas pressure, there is a sudden decrease in the amount of β-W phase at 1.4 Pa, then a further increase of working-gas pressure yields to the increase in the amount of b-W phase. The lattice parameters of alpha and beta tungsten film increase when the amount of a-W is increased, except when there is a high content of α-W (70 wt.%). The results of size-strain analysis, volume averaged grain size and r.m.s. microstrain obtained by different methods used in this work are in good agreement.

[1] J. Rodriguez-Carvajal, FULLPROF-A program for Rietveld Refinement, Laboratorie Leon Brillouin, CEA-Saclay, France (2000).
[2] R. J. Hill and C. J. Howard, *J. Appl. Cryst.* **20** (1987) 467-476.
[3] D. Balzar, *J. Appl. Cryst.* **25** (1992) 559-570.

s9.m29.p1 **A Structure Matching Algorithm for Problems Involving Z'>1 Structures.** Richard Ian Cooper, and David Watkin, *Chemical Crystallography, Dept of Chemistry, University of Oxford, UK. E-mail: richard.cooper@chemical-crystallography.oxford.ac.uk*

Keywords: Z prime>1; Structure matching; Analysis

Given two sets of corresponding atomic coordinates, there are several methods for calculating the best fit between them; a review of some is given by Watkin[1]. The aim is to calculate a transformation **D**, which rotates and translates a set of n three dimensional coordinates, $[x_{A1}, x_{A2}, ... , x_{An}]$ onto another set of coordinates, $[x_{B1}, x_{B2}, ... , x_{Bn}]$, while minimising a function of the difference between them.

However, all of these methods require that the atoms in one set are pre-sorted into an order corresponding to the order in which they appear in the other set; the atoms must have already been matched by a munificent user, so that, for example, the atom at x_{A3} should map onto the atom at x_{B3}. This task, of manually numbering or ordering fragments in an identical manner is wearisome and error-prone, and so we present: an algorithm for carrying out such a match automatically; an implementation of the algorithm within the structure analysis package CRYSTALS[2]; and some examples of its use in the routine processing of Z'>1 structures.

The algorithm is based on an analysis of each molecule's atomic types and bonding network to obtain a small initial pairing of key unique atoms. A three-dimensional best-fit calculation using just these paired atoms follows. The resulting transformation D_1 is applied and, subject to the constraints of the preceding bonding network analysis, unpaired atoms that lie closest together are matched up. A further three-dimensional best-fit calculation produces a more accurate transformation D_2.

The matched list of atoms may be used, for example, to produce a consistent numbering scheme for Z'>1 structures, requiring only the first molecule to be numbered manually. The transformation matrix, D_2, may be used to detect pseudo- or missing symmetry in a structure, or to produce diagrams of overlapping molecules in order to visualise the geometrical differences between them.

[1] Watkin, D. J. (1980) *Acta Cryst* A**36**, 975-978
[2] Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487