

MICROSTRUCTURE OF Al_3W THIN FILMS

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Origin of prominent texture that appeared in the $\text{Al}_{0.75}\text{W}_{0.25}$ thin film deposited on sapphire substrate, was disclosed by the SEM imaging of the film/substrate cross fracture. Columnar needle-like forms, about 100 nm in diameter, were frequently observed (Fig. 1a) protruding throughout the whole film thickness of 4 μm starting from the film/substrate interface to its surface. No such needle-like protrusions were found in the films deposited on monocrystalline Silicon substrate (Fig. 1b), neither in the $\text{Al}_{1-x}\text{W}_x$ films on sapphire for composition deviating from Al_3W , nor deposited at temperature outside the range of 500K-700K. Pronounced texture that was revealed in XRD experiment (Fig. 2) for this specific film, can certainly be related to the observed crystalline fibres.

Series of thin films with various composition $\text{Al}_{1-x}\text{W}_x$ ($0.20 < x < 0.33$) were deposited by magnetron sputtering (1) of target materials on a number of substrates (mono-Si, $\alpha\text{-Al}_2\text{O}_3$ sapphire wafers, ...) held at pre-selected temperature (300K – 700K) for a period of time to control the thickness (0.2 - 4 μm). Besides the SEM studies of local microstructure, the film's nanostructure and average crystal structure were investigated by XRD and GISAXS experiments (2).

All films deposited on both substrates at temperatures lower than 500K display XRD patterns (Fig. 2a) typical for amorphous or highly disorder nanocrystalline structure. In case of films on mono-Si substrate for deposition temperatures higher than 550K, the intensity and profiles of diffraction lines indicate increase of crystalline order with no traces of texture (Fig. 2c). In the special case of the $\text{Al}_{0.75}\text{W}_{0.25}$ film deposited at 500K - 700K onto $\alpha\text{-Al}_2\text{O}_3$ sapphire substrate, the azimuthal variation of diffraction intensity (Fig. 2b) reveals a degree of crystalline structure with preferred orientation; these features are more pronounced for higher film deposition temperature. Three V-marked spots in Fig. 2b reveal 1st, 2nd and 3rd order of diffraction from atomic layers with spacing $d=0.38\text{nm}$. Disk-like shape of these spots in reciprocal space corresponds with fibrous crystallite shape in direct space, and is therefore consistent with attribution of the texture to the needle-like crystals imaged in Fig. 1a. These spots ceased to exist in the XRD pattern of the film annealed at 1020K (Fig. 2d). All diffraction lines marked in Fig 2c can be assigned by the BCC lattice cell of tungsten: $a=0.316\text{ nm}$ (Fig. 3), indicating that it describes fairly well the average structure of the dominant phase in the $\text{Al}_{0.75}\text{W}_{0.25}$ film after annealing. Existence of an equilibrium Al_3W phase with such a simple crystal structure has not been reported so far, while a number of phases richer in aluminium (Al_4W , Al_5W , Al_{12}W) have been well established (3). However, for structural description of an indicative Al_3W phase, one can tentatively consider a larger cell (trigonal R-3m, $a\approx 0.632\text{nm}$; $\alpha\approx 90^\circ$), as the one schematized in Fig. 3b.

Prominent texture in XRD experiment (Fig. 2b&d) is consistent with the preferred crystallographic $\langle 111 \rangle$ orientation along the film normal (i.e. perpendicular to the film surface).

(1) N. Radić, B. Gržeta, O. Milat, J. Ivkov, M. Stubičar, Thin Solid Films 320 (1998) 192-197

(2) P. Dubček, N. Radić, O. Milat, NIM B 200 (2003), 329-332

(3) Binary Alloy Phase Diagram, ed. T. Massalski, ASM, Menlo Park, OH (1986), pp 179-181

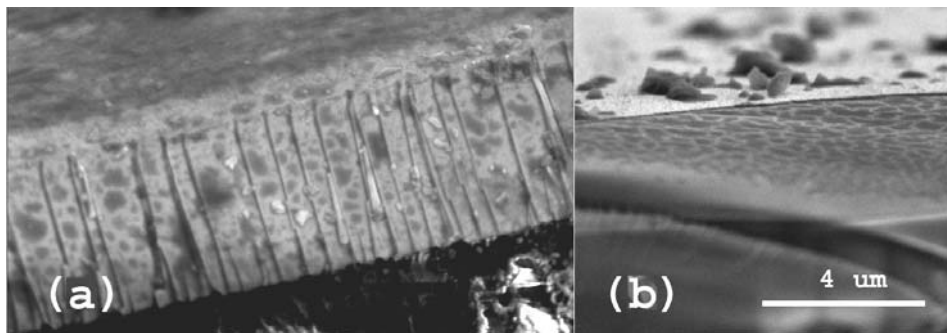


Fig.1. SEM images of cross fracture of $\text{Al}_{75}\text{W}_{25}$ film deposited on: a) sapphire; b) silicon substrate.

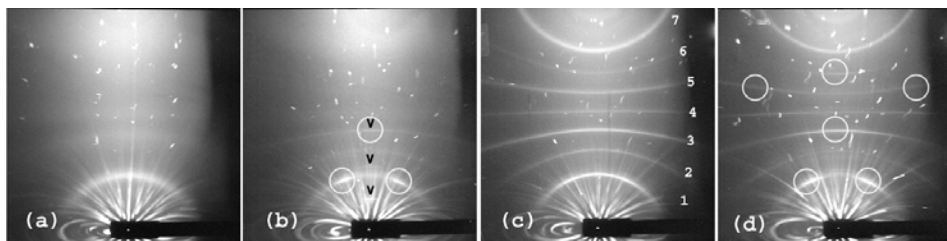


Fig. 2. XRD patterns of $\text{Al}_{75}\text{W}_{25}$ film deposited on: (a) silicon substrate at 400K; (b) sapphire substrate at 570K; (c) silicon substrate at 670K and annealed at 1020K; (d) sapphire substrate at 670K and annealed at 1020K. White radial streaks at the bottom and white circular spots overall are due to substrates. Regular circles mark maxima of diffraction lines intensity due to preferred crystallographic orientation; three V-marks in (b) point to spots of disk-like shape (discussed in the text); numbers at right in (c) mark the sequence of seven most prominent diffraction lines that can be assigned by the BCC cell of tungsten (see Fig. 3).

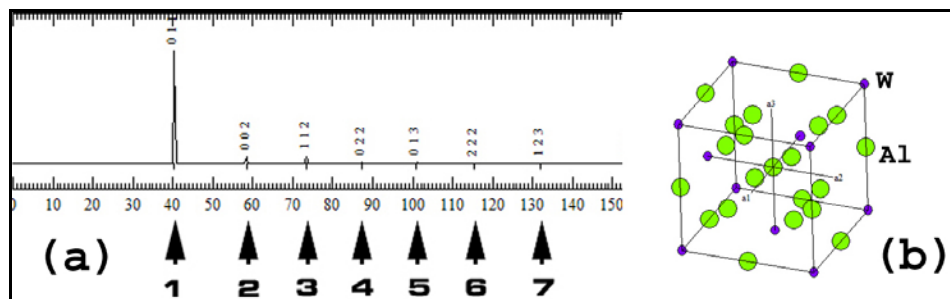


Fig. 3. (a) scheme of the XRD pattern as calculated for tungsten unit cell in the 2θ range of 0 – 150° in comparison with the positions (marked by arrows) of seven lines in Fig. 2. (b); trigonal superlattice cell for tentative description of average structure of an indicative Al_3W phase.