

## The growth and electrical properties of tantalum nanowires self-organized under the porous alumina film

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The development of material components at nanoscale whose structures exhibit novel physical and chemical properties has been the current trend in science and technology. As reported in our previous works [1,2], anodizing under certain conditions of sputter-deposited Ta-Al, Nb-Al, and Ti-Al metal bilayers results in formation of self-organized arrays of nano-sized anodic oxides of unique morphologies and complex chemical compositions. However, the detailed knowledge of these nanostructures is not currently complemented by a fundamental understanding of the growth and properties of self-organized network of valve-metal nanowires, which concurrently forms under the porous alumina film. In this paper, characteristic patterns of metal-oxide nanostructures derived from anodically oxidized Ta-Al bilayers have been prepared and characterized to obtain new insight into the growth and electric transport mechanism in the self-organized valve-metal nanowires.

Initial samples were tantalum films, 8 to 22 nm thick, and aluminum films, 200 nm thick, sequentially sputter-deposited onto dielectric substrates. The aluminium layers were potentiostatically anodized in oxalic acid solutions of various concentrations at anodic voltages ranged from 20 to 60 V to form porous-type anodic oxide films of different pore size and porosity. The tantalum underlayers were then locally anodized through the nano-sized alumina pores. In order to examine the effect of higher formation voltages on the residual tantalum thickness, the specimens were re-anodized in 0.5 M boric acid solution to different final voltages in the range of 53 - 90 V. The development of tantalum nano-network was studied by SEM, TEM, XPS, and EDX point spectroscopy. The electro-physical properties of the oxide parts of the anodic films were examined by electrochemical impedance spectroscopy (EIS) in 0.5M H<sub>3</sub>BO<sub>3</sub>/0.05M Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub> solution at 100 mV of sinusoidal voltage in the frequency range of 50 mHz - 2 MHz. The behaviors of sheet resistance (SR) and temperature coefficient of resistance (TCR) of the films were determined in a wide temperature and frequency range.

It was found that the films are composed of multiplied oxide hillocks closely adjusted to, but separated from, each other by unanodized tantalum metal, which is self-organized in the conductive network, as shown in fig. 1 (black parts). A single built element (tantalum nanowire) can be 5 - 15 nm thick and 15 - 80 nm long while the average size of the

oxide hillocks incorporated in the tantalum nano-network ranges from 25 to 90 nm. The oxide hillocks have mixed composition of non-stoichiometric tantalum sub-oxides, alumina, and electrolyte-derived species. The ionic transport mechanism for the Ta<sup>5+</sup>, Al<sup>3+</sup> and O<sup>2-</sup> ions during the oxide growth is discussed in this work. The net-like structure and 3D nanoscale topography of the residual tantalum metal result in a wide range of possible SR values (10<sup>2</sup>-10<sup>6</sup> Ohm/sq) depending on the formation voltage. The TCR values are negative and change between 350 and 10 ppm/K proportionally to physical size of the oxide hillocks. This implies an increased transition to hopping or tunneling conduction at elevated temperatures. The behavior observed can be explained by considering the films as periodical heterogeneous two-phase (metal-insulator or metal-semiconductor) systems with impurity depth and width profiles at nanoscale. Further, oscillations of the dc conductance observed with increasing the forming voltage even at room temperature can be attributed to the quantization of the electron energy into discrete sub-bands as a result of the confinement due to the formation of a symmetrical quantum well from the tantalum-tantalum network clad with the anodic alumina film.

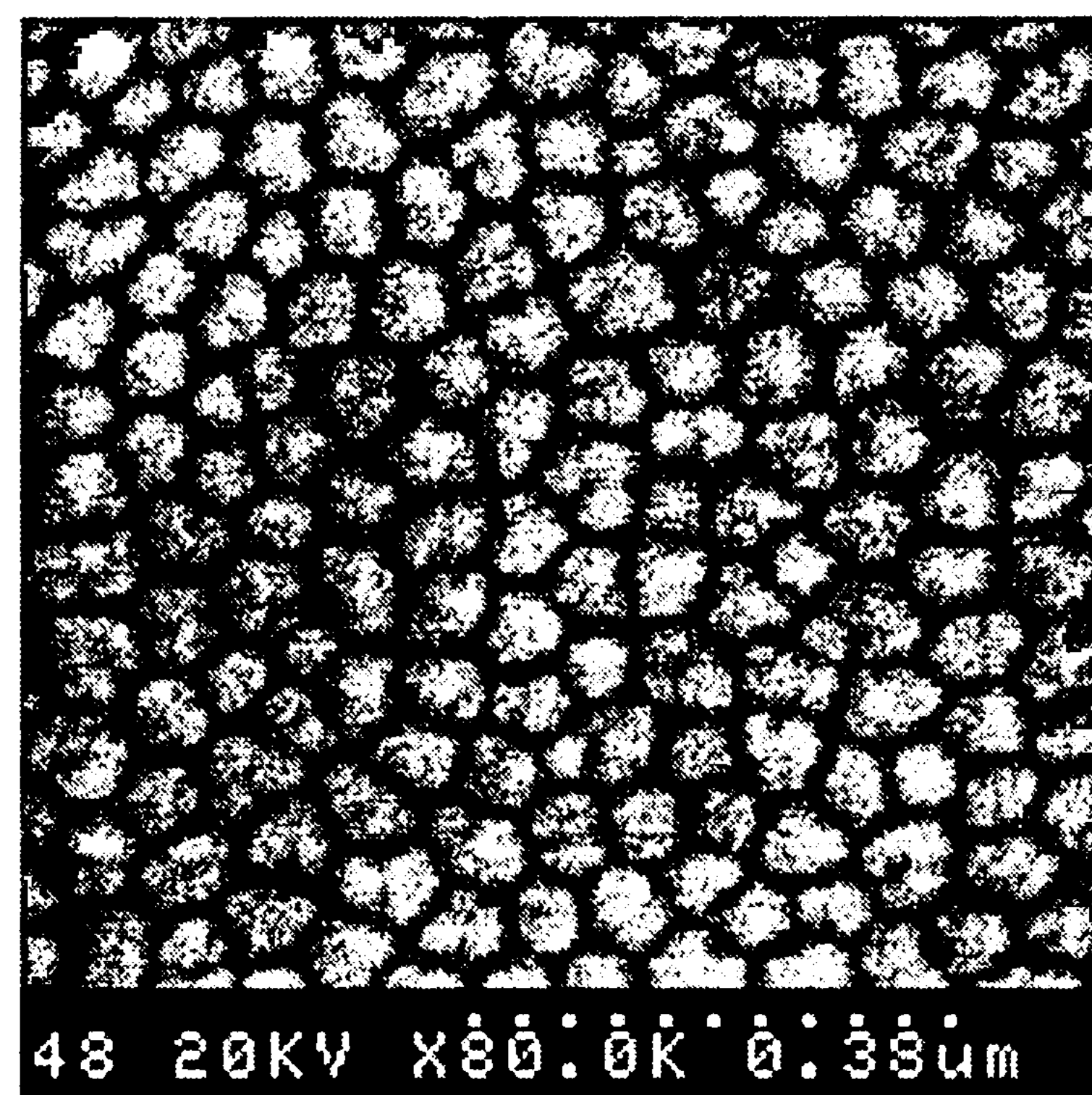


Fig. 1. Typical SEM surface view of the self-organized nano-network derived from an anodically oxidized Ta-Al metal bilayer

The new findings are valuable for fabricating integral thin-film resistors with significantly improved performances and, more generally, as models for studying field-assisted ionic transport and quantum-size effects in the nano-structured metal-oxide anodic films.