2012 Graduate Research Symposium
Department of Mechanical Engineering, Iowa State University
March 29, 2012

Transient Thermal Characterization of Single Anatase TiO_2 nanowire with secondary porous structure

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Introduction

Single anatase TiO_2 nanowire is synthesized using electrospinning technique and is then bridged over a pre-processed 100 μm-wide TEM grid for thermal characterization. Transient electrothermal method is applied with proper calibration process for determining major thermophysical properties. SEM diagnostics of the sample show that calcinations for crystal structure transition from amorphous to anatase has induced distinct porous surface structure. This porous structure implies interior porous structure and lower density. Also, due the reduced density and enhanced grain boundary scattering, phonon transport is hindered and therefore the thermal conductivity is measured to be lower than value of bulk counterpart. Furthermore, nonlinear effects are also examined and further analyzed to acquire more precise thermophysical properties.

Sample Preparation: Electro-spinning method[1] is applied to deposition TiO_2 nanowires. A specially-processed TEM grid is used as collector. Due to the coating nature on the silicon TEM grid, one slot is left to provide symmetric electrical force to align the deposited TiO_2 nanowires, as shown in Fig. 1. Sputter coating with iridium and FIB-assisted Pt soldering are used to enhance the thermal and electrical contact between the nanowire and grid surface. Even though, unexpected effects such as capacitance effect still appear in the data. In addition, porous surface feature is observed after the amorphous TiO_2 transits into anatase during the calcinates, as shown by the SEM images.

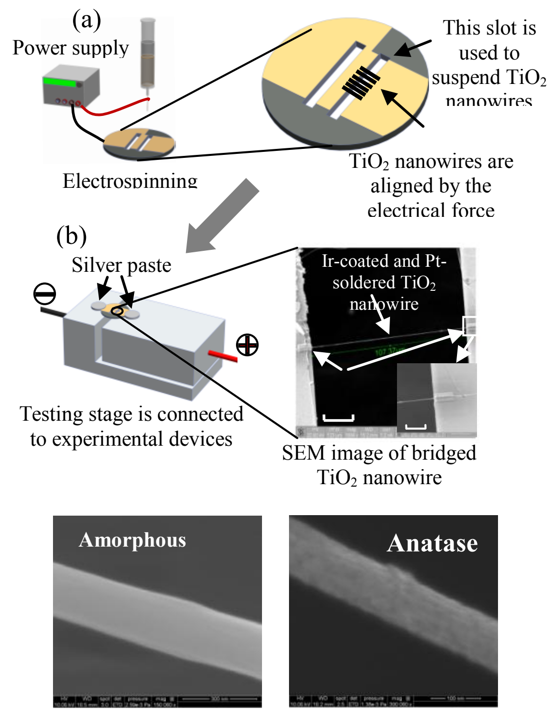

Fig.1. (a) AFM images of nanostructure of TiO_2 thin film: (left) before annealing; (right) after annealing.

Experiment method: TET is transient technique to obtain signal strong enough in less than a second. In this technique, 1-D heat transfer model is presumed and non-constant joule heating is introduced to induce resistance change. The theoretical temperature variation is,

$$T(t) = T_0 + \frac{8q_0L^2}{k\pi^4}\sum_{m=1}^{\infty}\frac{1-exp\left[-(2m-1)^2\pi^2\alpha t/L^2\right]}{(2m-1)^4} \quad (1)$$

Fig.2 shows a typical *U-t* profile recorded by the oscilloscope. The rising phase is followed by a steady state which indicates the heat transfer equilibrium. The

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transient state during the rising phase is fitted to determine the thermal diffusivity. The overall temperature change can be further used for derivation of thermal conductivity based on calibration process.

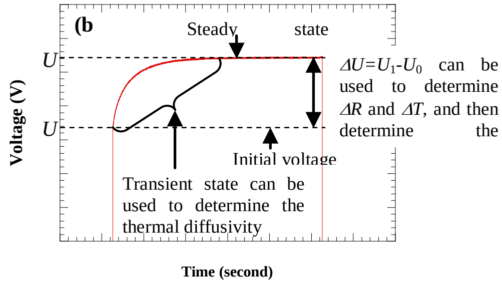

Fig.2 Typical U-t profile including both transient stat e and steady state.

Nonlinear effects: Complicated preparation process inevitably introduces nonlinear effects as shown in Figure 3. On left is a linear temperature rise and on the right, because of nonlinear effects, the temperature increases gradually and then cause ambiguity to determine the starting point of linear Ohm's effect.

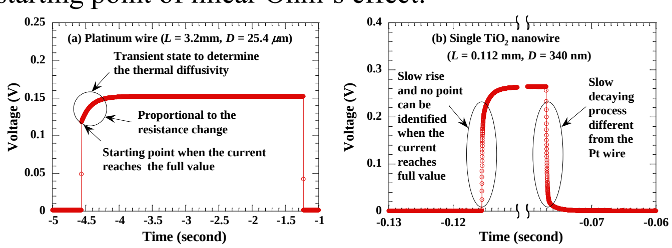

Fig.3 Comparison between U-t profiles with linear effect (left) and nonlinear effect (right).

Two methods are derived to suppress the impact of nonlinear effects: generalized function analysis (eq. (2)) and direct derivation method (Eq. (3)).

$$\frac{U_1}{U_2}=\frac{I_{01}}{I_{02}}\left[1+f_2(I_1^{\,2}-I_2^{\,2})\right] \quad (2)$$

$$R(t)=\frac{U}{I_0-CdU/dt} \quad (3)$$

Results and Discussions: The porous surface (secondary surface porosity) demonstrates significant influence on thermophysical properties. Both density and thermal conductivity show strong reduction because the porous structure and calcinations-induced crystal cluster. Phonon-vacancy and phonon-grain boundary scattering are two main mechanisms that account for the distinct reduction, as seen in Fig. 4.

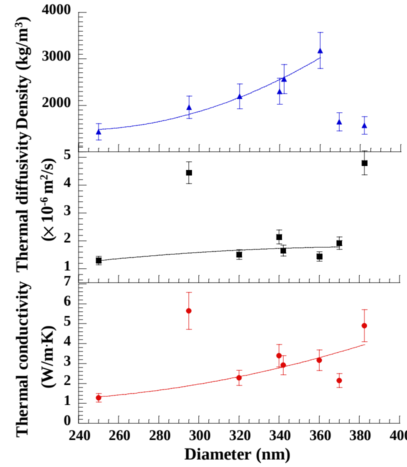

Fig. 4 Comparison between normalized temperature rise versus time between theoretical and experimental data for the samples.

In addition, strong connections between diameters and thermoaphysical properties are observed in Fig. 4 as well. Both density and thermal conductivity increases when diameter increases. Reasonable interpretation of this tendency is that larger diameters features more aggregated crystal cluster and consequently contains less porous structure. Also, larger diameter implies lower phonon-boundary scattering which in turn contributes to higher thermal conductivity.

Conclusion: For the first time, single TiO_2 nanowire, the basic building blocks of meso-scopic structures, were fabricated using an electrospinning technique and interior interconnected TiO_2 nanofibers were confirmed by AFM images. The TET technique was adopted to provide a full spectrum measurement of physical properties of the nanowire along with imaging of its porous features. Thermal conductivity and density presents strong dependence on the nanowire diameters, demonstrating that confinement from boundary and interior void space significantly hinders phonon transport.

Acknowledgements: Support of this work from the National Science Foundation (CBET-0931290) is gratefully acknowledged.

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