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Supporting Information for

Partially Crystallized TiO_2 for Microwave Absorption

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1. Experimental details: Titanium sheets (99.96%, 0.5 mm thick) were ground and polished to remove the oxide film. After ultrasonically degreased in acetone, methanol, and deionized water, Ti substrates were dried in nitrogen, and used as the anode. A platinum foil served as the cathode with a distance of 2 cm from the anode. Square-shaped Ti foils (1 × 1 cm) were exposed to the electrolyte. Anodization was performed in 150 ml ethylene glycol (99.8%, Sigma-Aldrich) containing 0.25 wt.% NH_4F (98%, Sigma-Aldrich) and 2 vol.% deionized water under 60 voltages for 10 hours. To avoid cavities in TiO_2, the as-prepared samples were kept in ethanol at 30°C for 48 h to eliminate inner stress caused by the generation of oxide. The resultant oxide was rinsed with methanol and dried in air. The abrupt annealing was carried out by directly putting the as-anodized TiO_2 in pre-heated furnace of controlled temperatures for 1 h.

2. Characterization: (1) The X-ray diffraction spectra (XRD) were acquired on a Bruker D2 Phaser X-ray diffractometer. (2) The Raman spectra were collected on an Renishaw RM1000 Research Laser Raman spectrometer. (3) In-situ synchrotron HT-XRD was conducted at Australian

Synchrotron. 50 wt. % standard Al_2O_3 was added into anodic TiO_2, acting as the reference for further analysis. (4) Morphological analyses of specimens were performed on a field-emission scanning electron microscope (FEI Quanta 200F ESEM). The cross-sectional morphologies of nanotubes along their depth were viewed at the fracture part. (5) The transmission electron microscopy (HRTEM) was taken on Tecnai TF20 TEM. (6) The elemental composition was measured by Kratos Axis UltraDLD X-ray Photoelectron Spectrometer (XPS). (7) Electron spin resonance spectroscopy (ESR) was recorded at 300K and 100K temperatures on a JEOL - JES-FA200 Electron Spin Resonance Spectrometry. (8) The complex permittivity and permeability of samples were measured at the frequency range of 2–18 GHz using a N5244A PNA-X Microwave Network Analyzer. The ground powders were dispersed in melting paraffin wax, and the mixture was cast into a ring mold with thickness of 2.0 mm, inner diameter of 3 mm, and outer diameter of 7 mm. The contents of TiO_2 nanocrystals were 40 wt%, 60 wt% and 80 wt%, and the testing was performed at room temperature. (9) Thermal diffusivity of anodic TiO_2 was measured using NETZSCH LFA 447 Nanoflash at room temperature. Stripped oxide films were ground into fine powders, and dispersed in melted paraffin at concentrations of 60 wt.%, which was then made into disk shape.

3. Supporting Figures

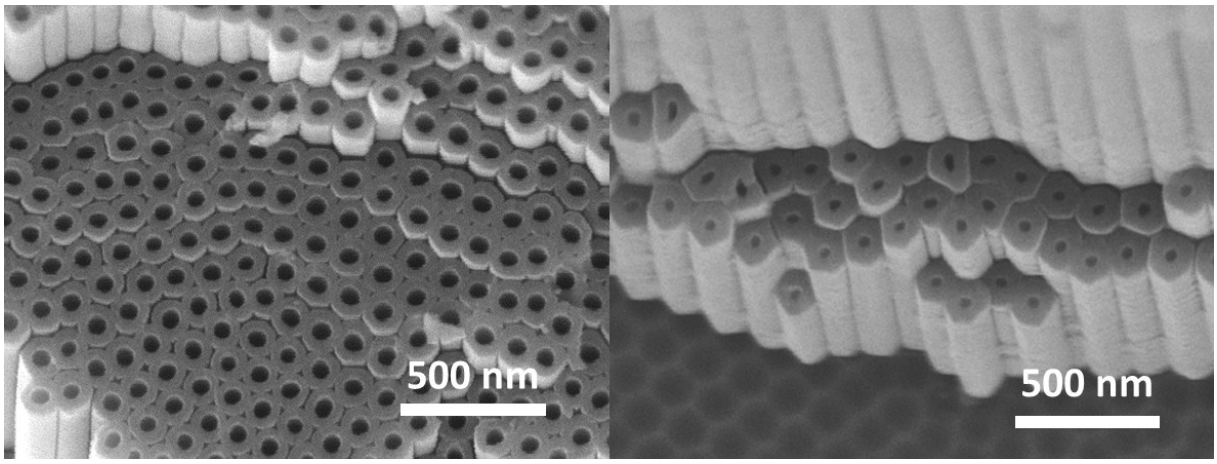

Figure S1. SEM images of as-anodized TiO_2

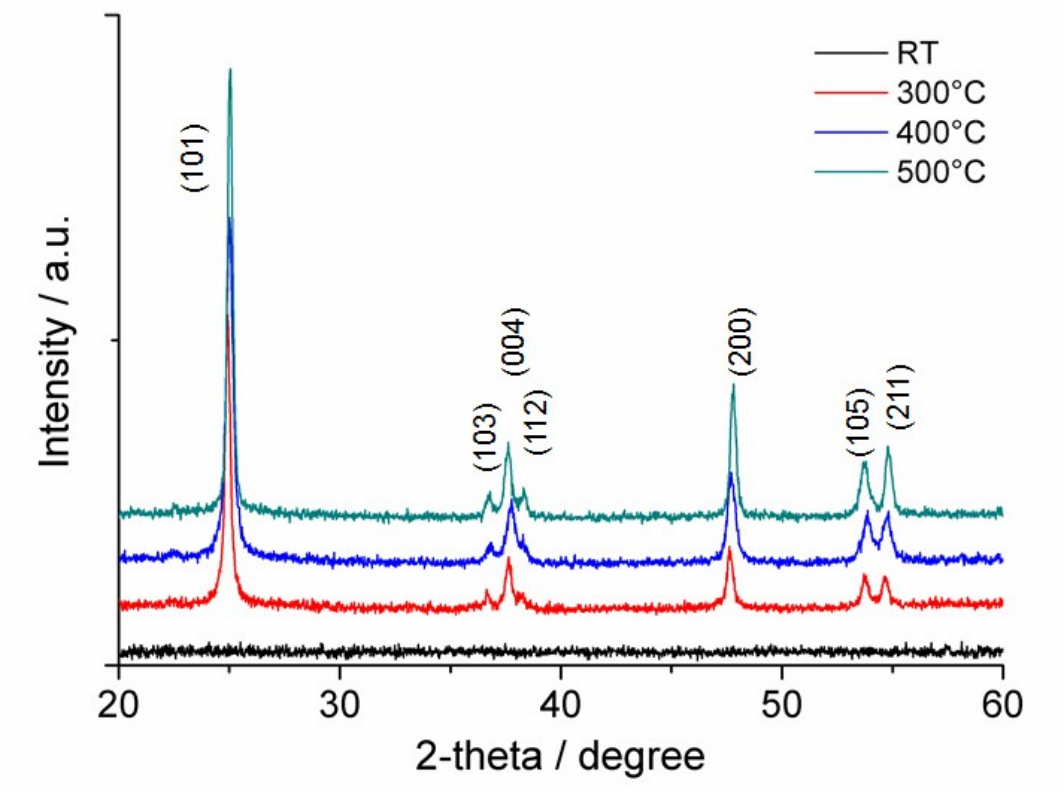

Figure S2. X-ray diffraction of anodic TiO_2

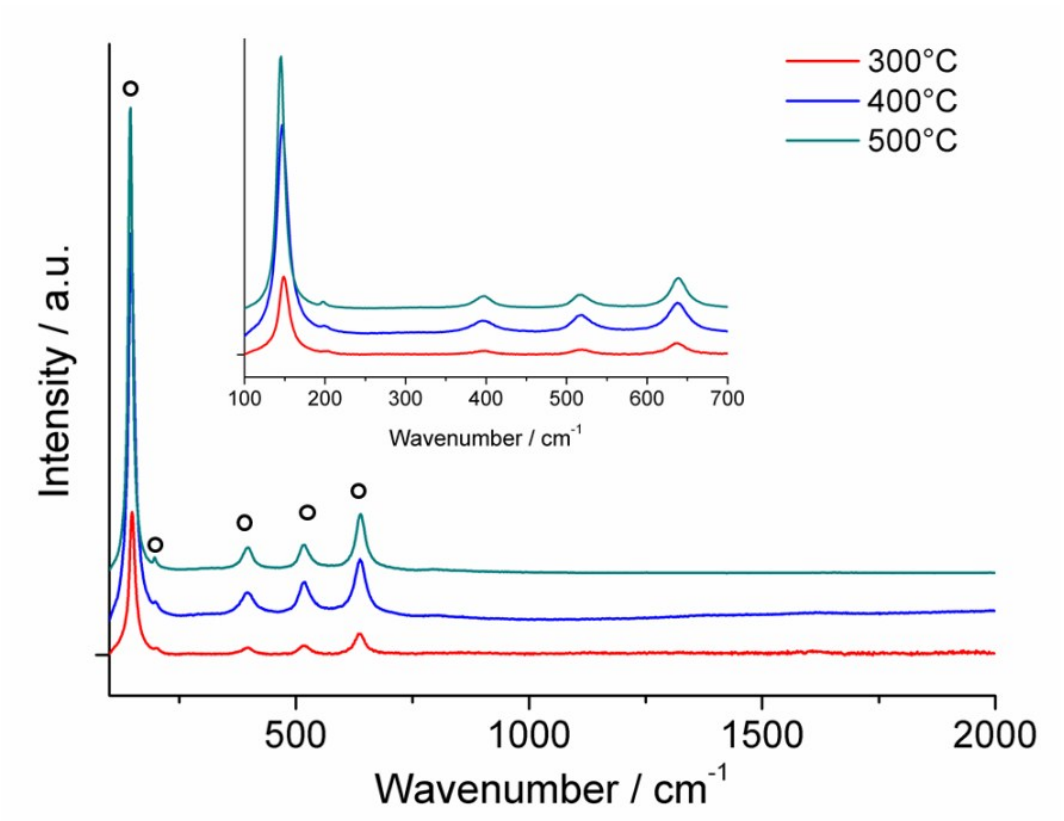

Figure S3. Raman Spectra of anodic TiO_2

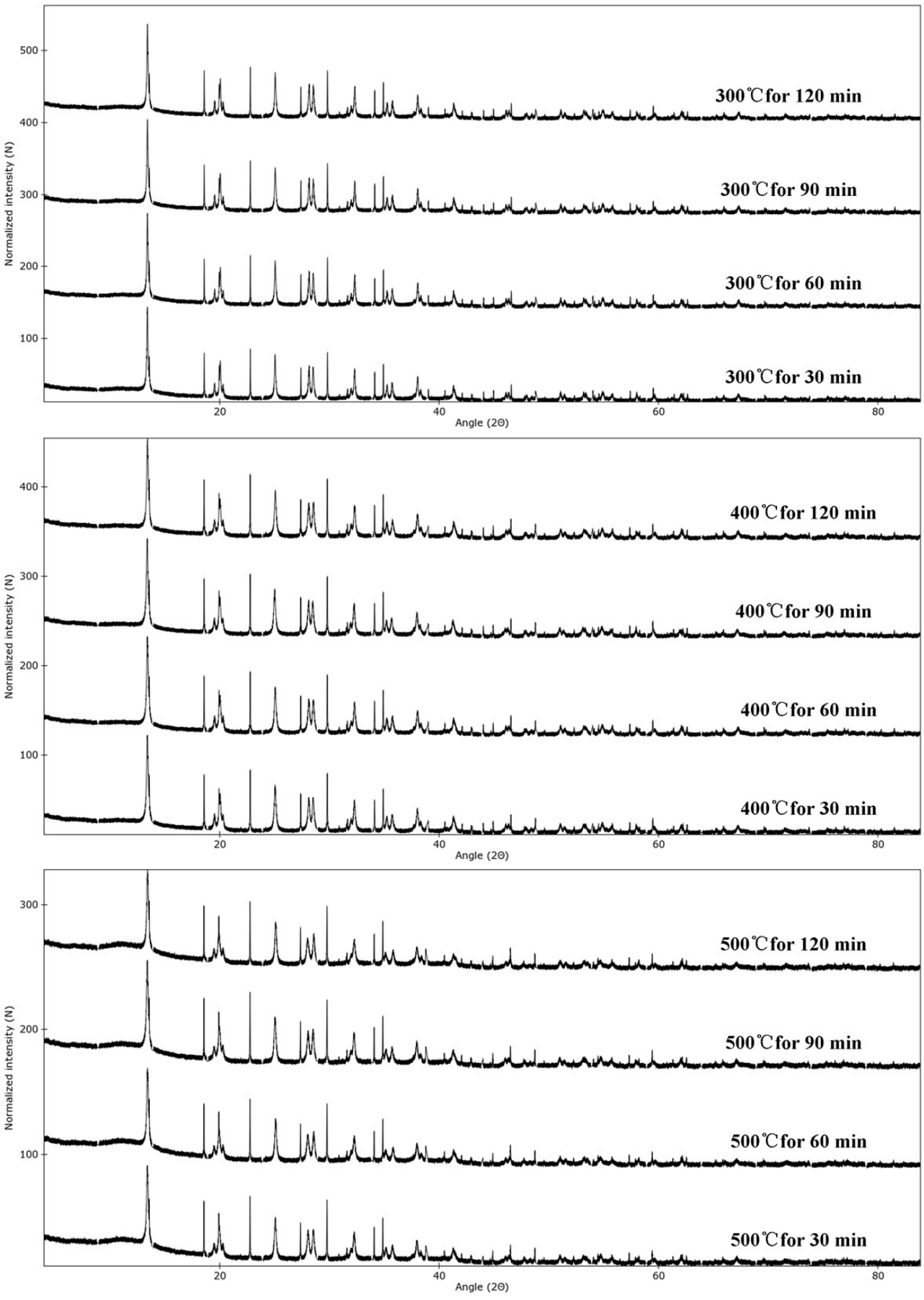

Figure S4 Synchrotron HT-XRD of the mixture of anodic TiO_2 & standard Al_2O_3

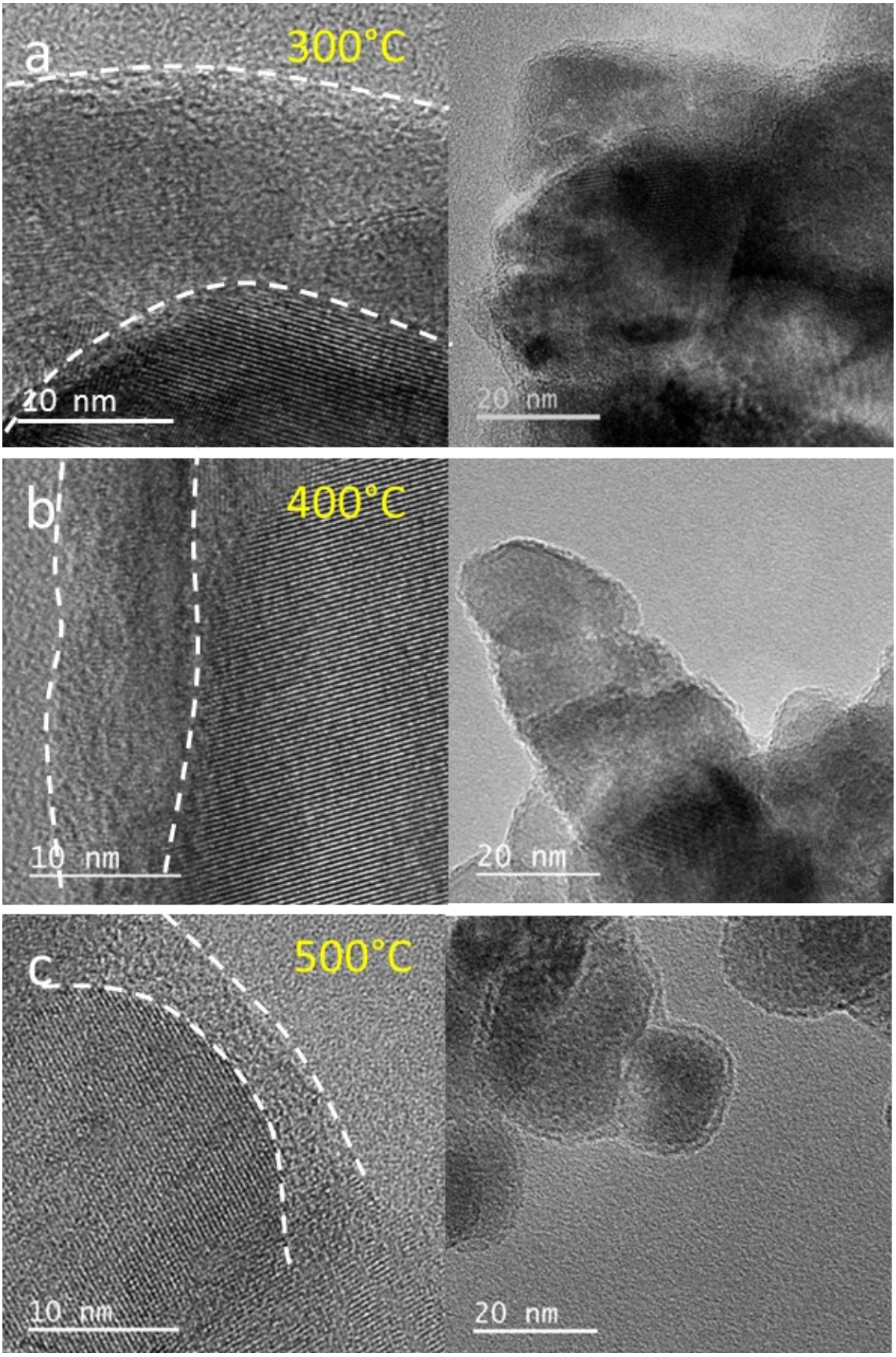

Figure S5. TEM images of the inner nanocrystals of anodic TiO_2 annealed at different temperatures.

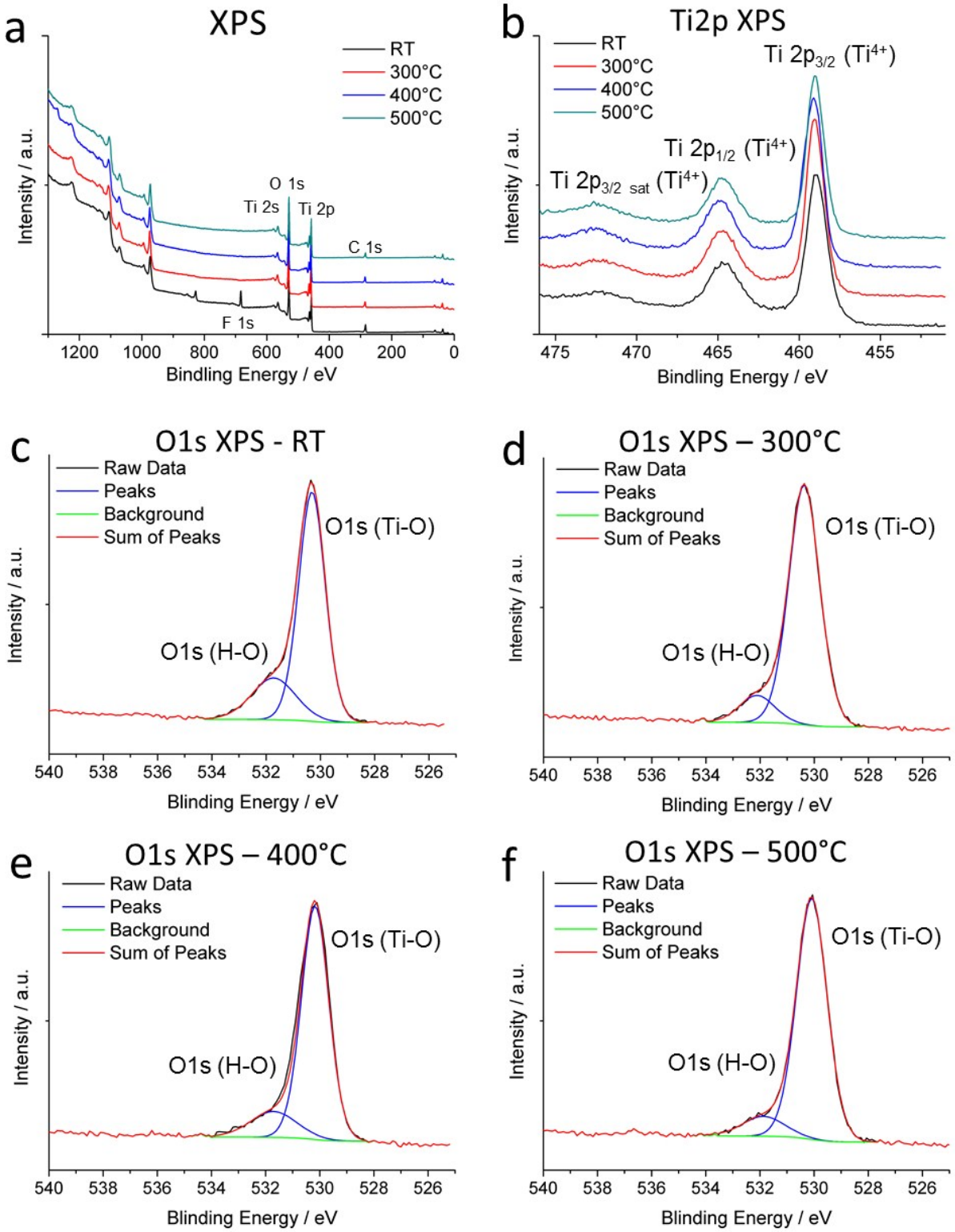

Figure S6. XPS of anodic TiO_2

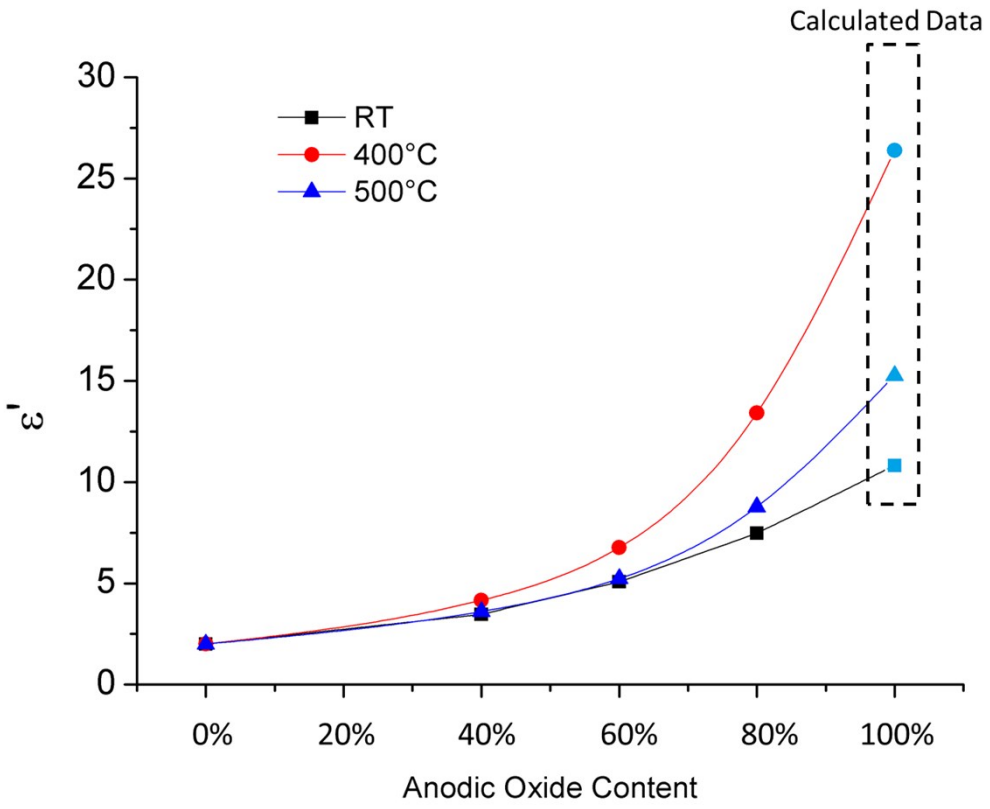

Figure S7 Real values of anodic TiO_2 complex permittivity. The fitting equations for the curves are:

Eq. 1 for Fitting Curve (RT): $y= 2.09984 + 1.01017* X + 4.90877* X^2+ 2.78114*X^3$

Eq. 2 for Fitting Curve (400°C): $y= 2.09984 + 11.125* X -33.64083* X^2+ 46.77595*X^3$

Eq. 3 for Fitting Curve (500°C): $y= 2.09984 + 5.86991* X -13.57964* X^2+ 20.85993*X^3$

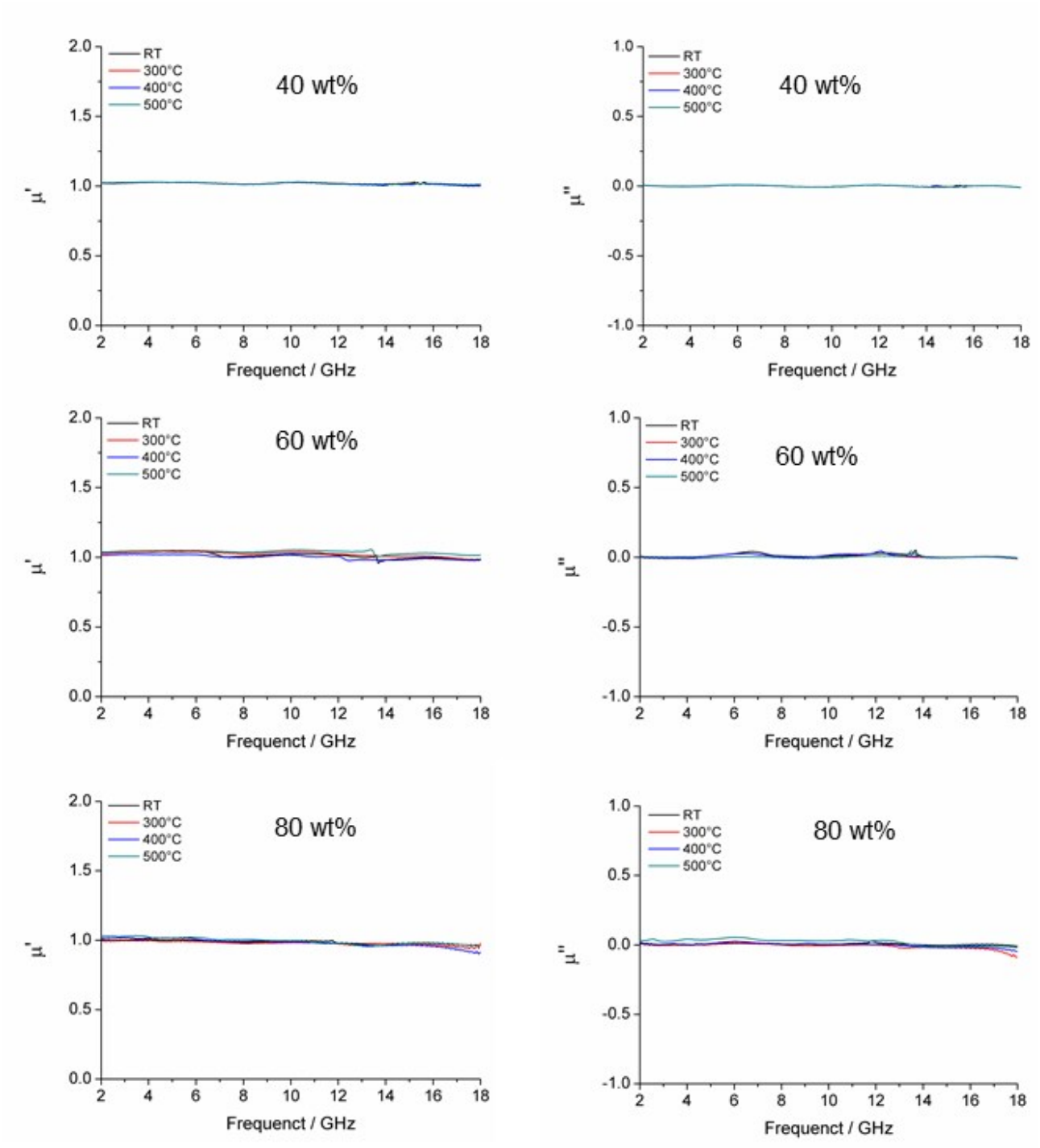

Figure S8 Real and imaginary values of anodic TiO_2 complex permeability

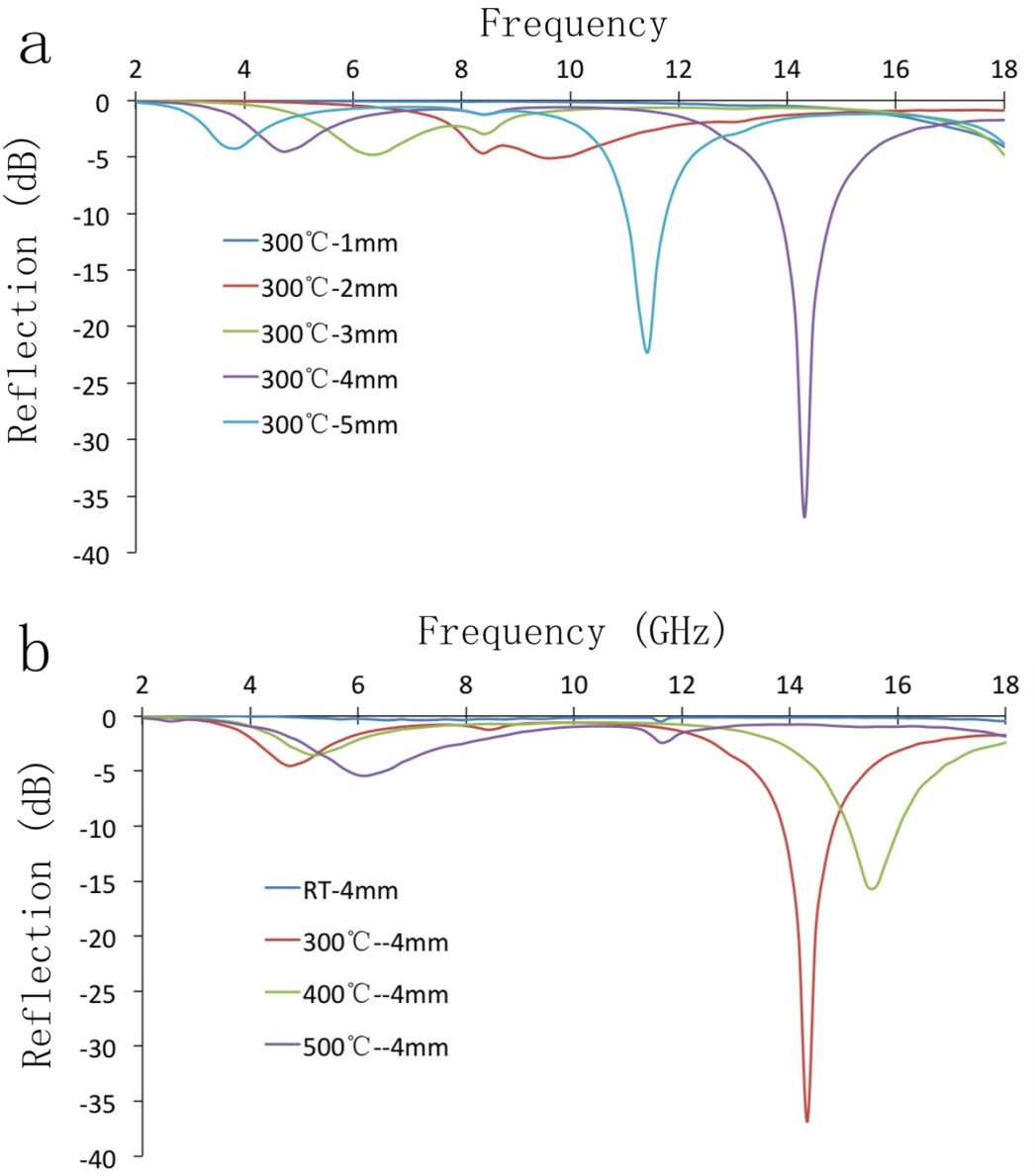

Figure S9 (a) Influence of coating thickness on microwave absorption; (b) Influence of heat treatments on microwave absorption.

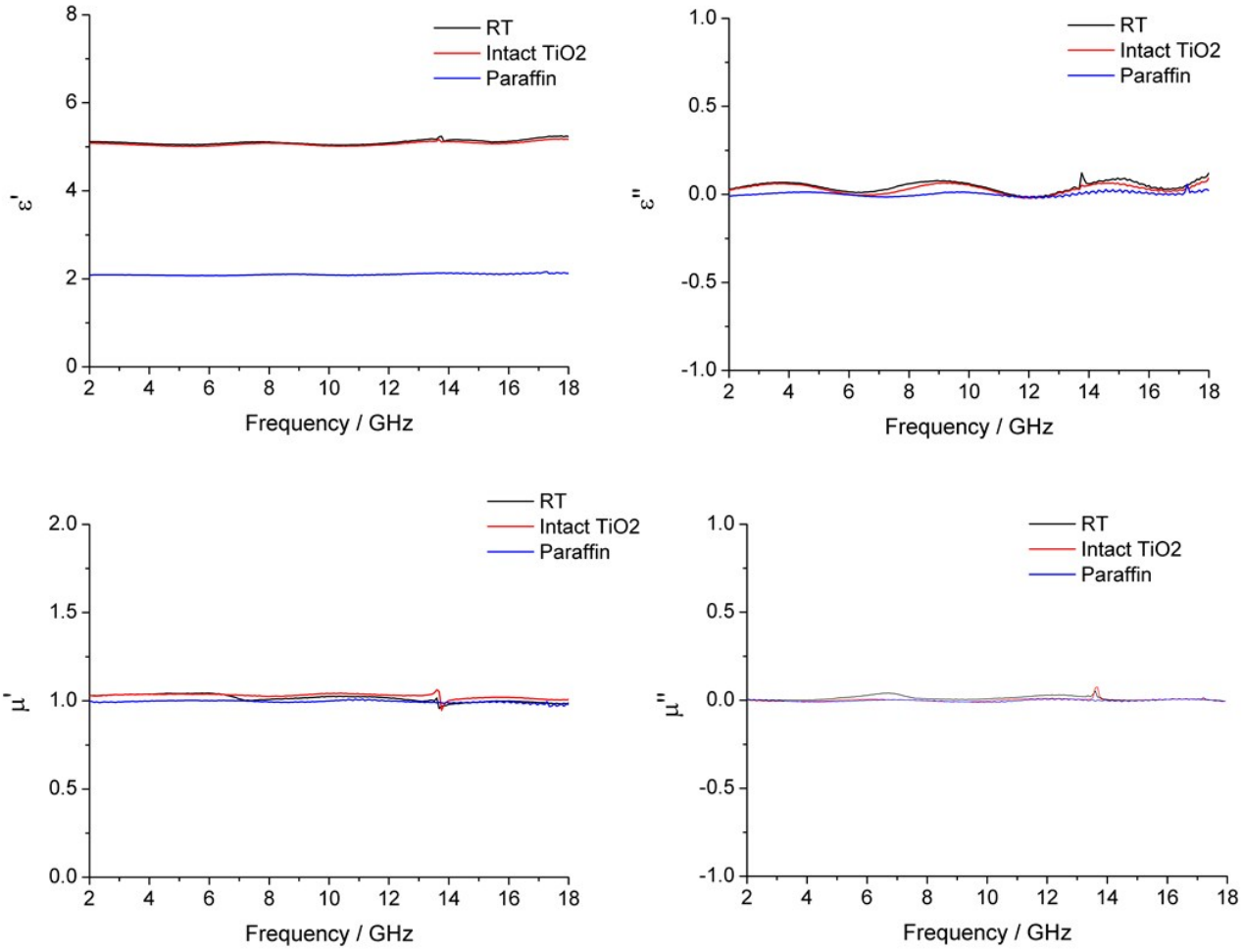

Figure S10. Real and imaginary values of the complex permittivity and permeability of intact TiO_2 (60 wt%), as-anodized TiO_2 (60 wt%) and pure paraffin.

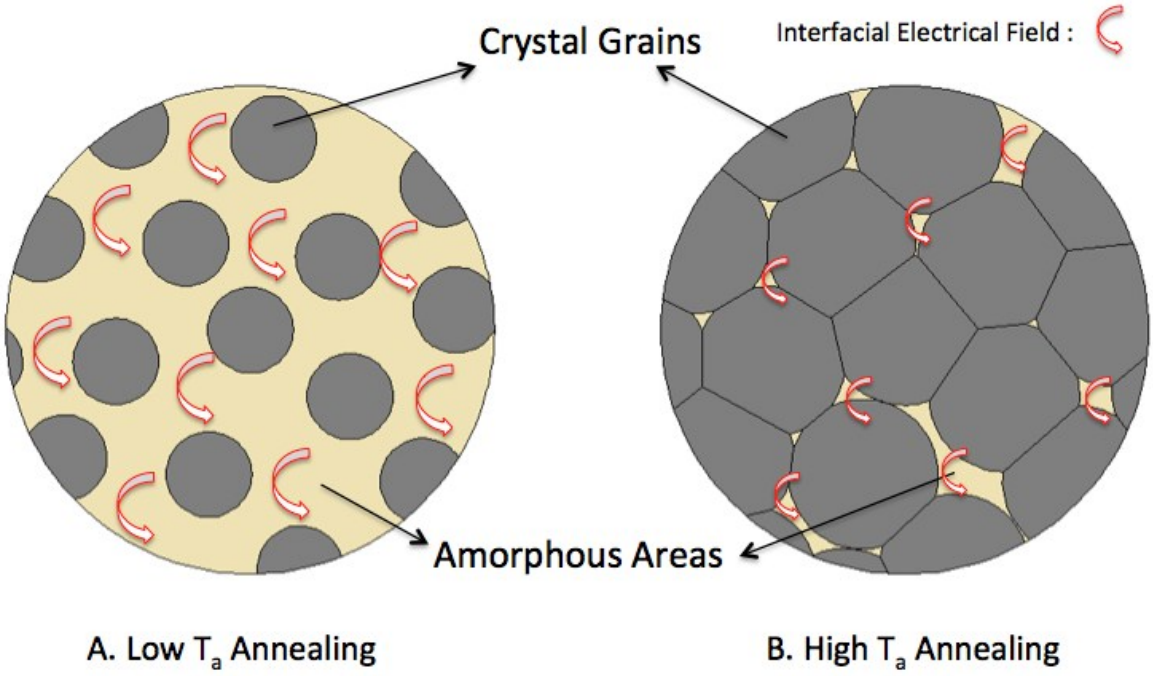

Figure S11 Schematic diagram for partially crystalized TiO_2

4. Supporting Table

Samples	Pure paraffin	As-anodized TiO_2	300°C	400°C	500°C
Thermal diffusivity (mm^2/s)	0.117	0.198	0.228	0.233	0.253
Thermal Conductivity(W/(m K))	0.173	0.499	0.588	0.626	0.675

Table. S1 Thermal diffusivity and conductivity of 60 wt.% anodic TiO_2 with 40 wt.% paraffin wax