

Three-dimensional SiO₂@Fe₃O₄ core/shell nanorod array/graphene architectures: synthesis and electromagnetic absorption property

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Experimental section

Materials. Graphene used in this work were purchased from Nanjing XFNano Material Tech Co., Ltd, its thickness and surface area are about 0.8 nm and 500-600 m^2g^{-1} , respectively. The oxygen content in the graphene is about 7wt%. The synthesized processes of graphene is described below. Typically, graphite oxide (GO) was prepared from natural graphite through a modified Hummers method.^{S1} The GO was thermally treated at a high temperature under N_2 flow, and then was reduced at 1200 °C under H_2 atmosphere. All of other reagents were analytically pure, and purchased and used without further purification.

Growth of β -FeOOH seeds on the graphene sheets. 0.01 g of the graphene was dispersed into 300 mL water, and then 4.0 g of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ was added. The mixture above was kept at 50°C for 2 h under stirring. The precipitates were separated by centrifugation, washed with distilled water and absolute ethanol, dried under vacuum.

Synthesis of 3D β -FeOOH nanorod array/G architecture. 15 mg of the above product was dispersed in 35 mL of the solution containing 0.5 g of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and 1.0 g of sodium nitrate under stirring for 20 min. The mixture above was then transferred into a Teflon-lined stainless steel autoclave with a capacity of 50 mL for hydrothermal treatment at 60°C for 12 h. As the autoclave cooled to room temperature naturally, the precipitates were separated by centrifugation, washed with distilled water and absolute ethanol, and dried in air.

Synthesis of 3D SiO_2 @ β -FeOOH nanorod array/G architecture. 0.07 g of 3D β -FeOOH nanorod array/G architecture were dispersed in 80 mL 2-propanol, Under continuous mechanical stirring, 8 mL deionized water, 2 mL ammonia solution and 0.25 mL tetraethyl orthosilicate were consecutively added to the dispersed solution.

The reaction was kept for 4 h under stirring at room temperature. The precipitate was washed several times with deionized water and anhydrous ethanol, respectively, and then dried at room temperature.

Fabrication of 3D SiO₂@Fe₃O₄ nanorod array/G architecture. 3D SiO₂@Fe₃O₄ nanorod array/G architecture was fabricated by thermal treatment of the 3D SiO₂@β-FeOOH nanorod array/G architectures at 280°C for 3 h in H₂/Ar flow.

Structural Characterization.

The morphology and size of the synthesized samples were characterized by scanning electron microscope (SEM) [HITACHI S-5200] and an FEI Tecnai-F20 transmission electron microscope (TEM) equipped with a Gatan imaging filter (GIF). The operating voltages of SEM and TEM are 5 and 200 kV, respectively. The crystal structure of the sample was determined by X-ray diffraction (XRD) [D/max 2550 V, Cu KR radiation]. X-ray photoelectron spectroscopy (XPS) measurements were carried out using a spectrometer with Mg Ka radiation (ESCALAB 250, Thermofisher Co.). The magnetic property of the product was measured by a vibrating sample magnetometer (VSM).

EM parameter measurements.

The EM parameters of samples were measured by using the transmission/reflection coaxial line method. The measurement setup consisted of in an ANRITSU 37269D vector network analyzer with a synthesized sweep oscillator source and an S-parameter test set. The measured samples were prepared by uniformly mixing 20 wt% of the sample with a paraffin matrix. The mixture was then pressed into toroidal shaped samples (φ_{out} : 7.00 mm; φ_{in} : 3.04 mm).

Table S1 The comparison of the minimal R_L between magnetic materials and 3D architecture.

Materials	$R_{L, \min}$ (dB)	C (wt%)	Reference
$\text{Fe}_3\text{O}_4/\text{ZnO}$	-30.0	50	42
$\text{Fe}_3\text{O}_4/\text{TiO}_2$	-20.6	50	43
$\text{Fe}_3\text{O}_4/\text{SnO}_2$	-27.4	80	40
$\text{Fe}_3\text{O}_4/\text{C}$	-27.4	55	44
3D architecture	-31.9	20	This work

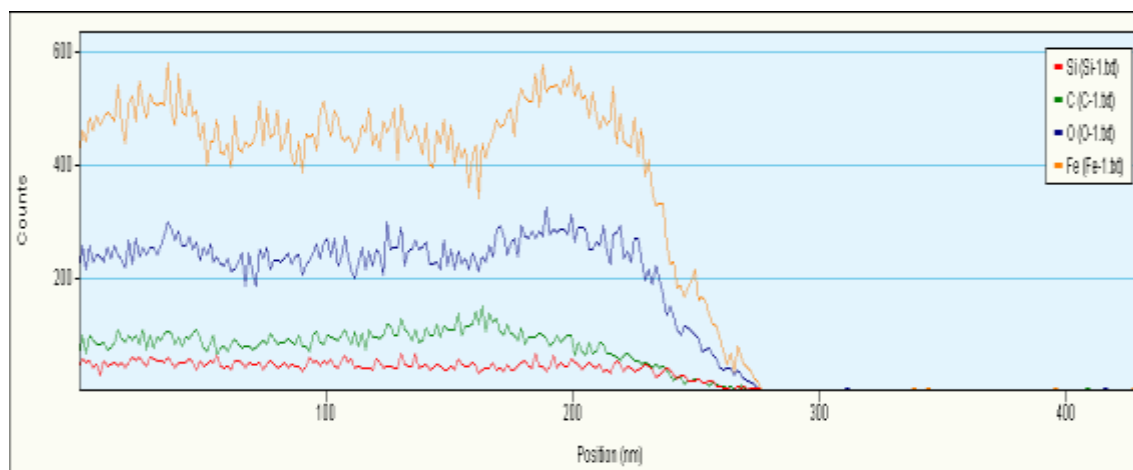
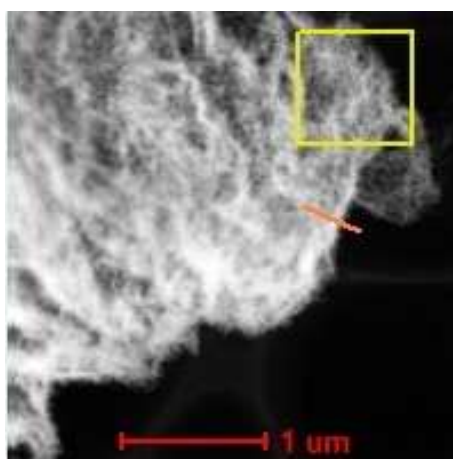


Figure S1 HAADF-STEM image of 3D $\text{SiO}_2@ \beta\text{-FeOOH}$ core/shell nanorod array/G architecture and Line scanning profiles along the red line in STEM image.

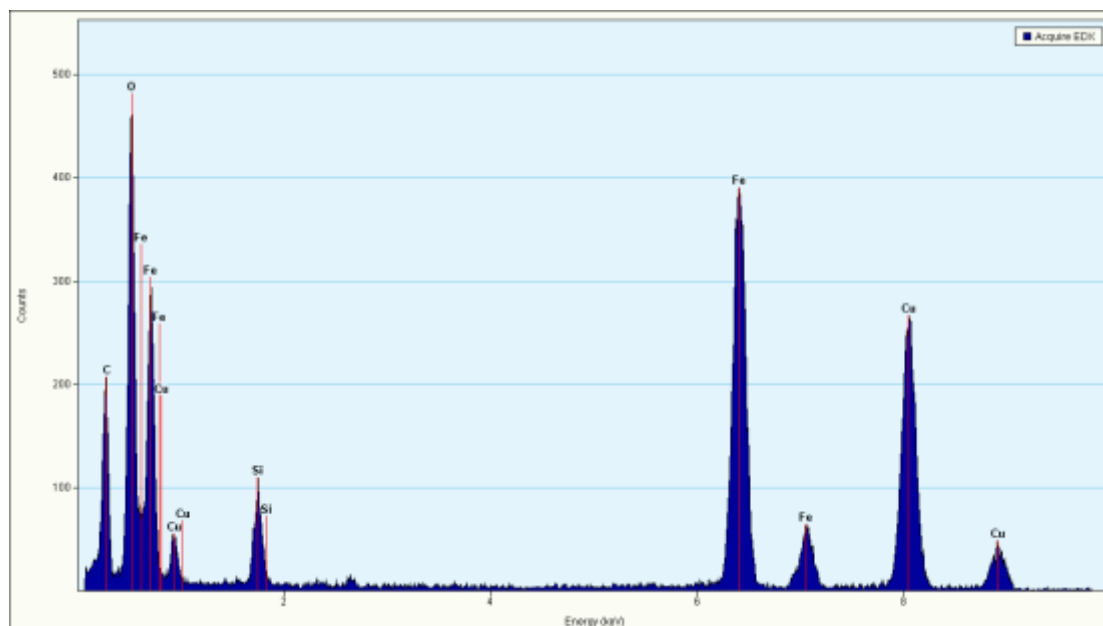


Figure S2 EDX pattern of 3D SiO₂@ β -FeOOH core/shell nanorod array/G architecture

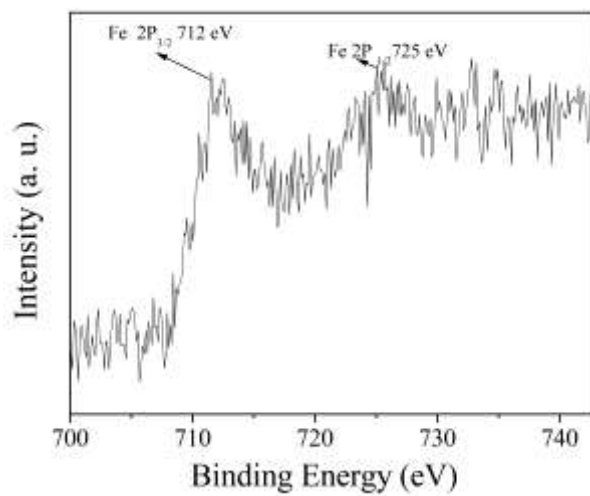


Figure S3 Fe2p core level XPS spectrum of the D SiO₂@Fe₃O₄ core/shell nanorod array/G architecture

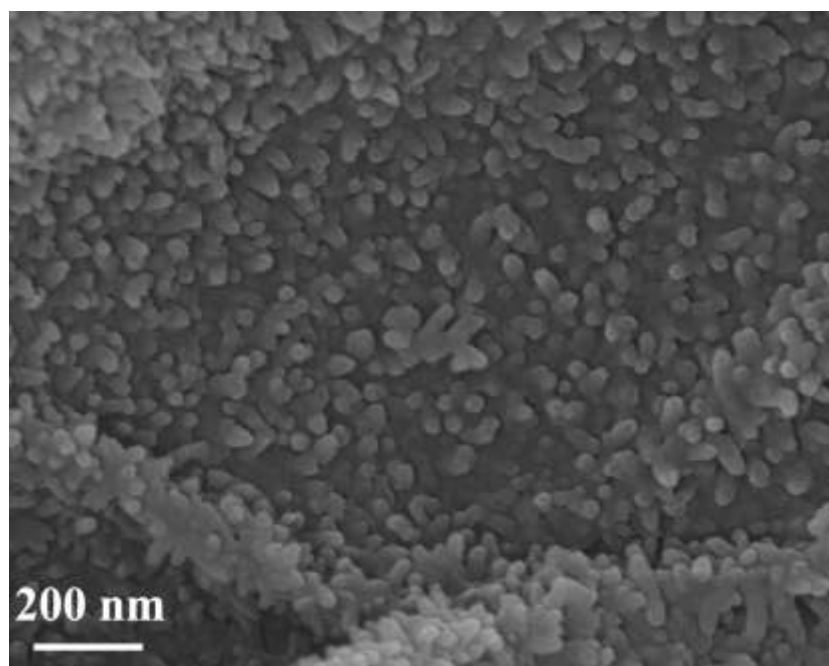


Figure S4 SEM image of the D SiO₂@Fe₃O₄ core/shell nanorod array/G architecture

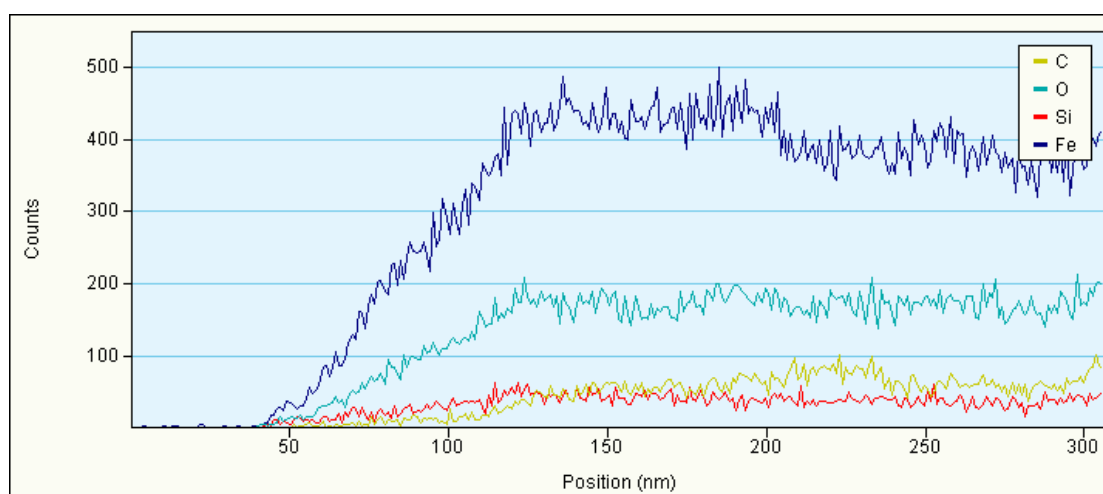
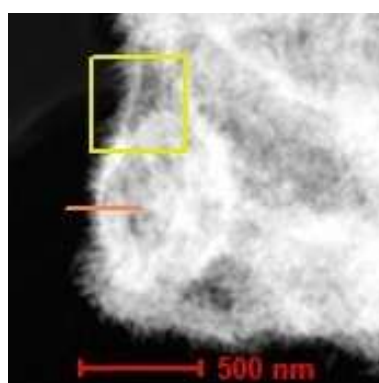


Figure S5 HAADF-STEM image of the D SiO₂@Fe₃O₄ core/shell nanorod array/G architecture and Line scanning profiles along the red line in STEM image.

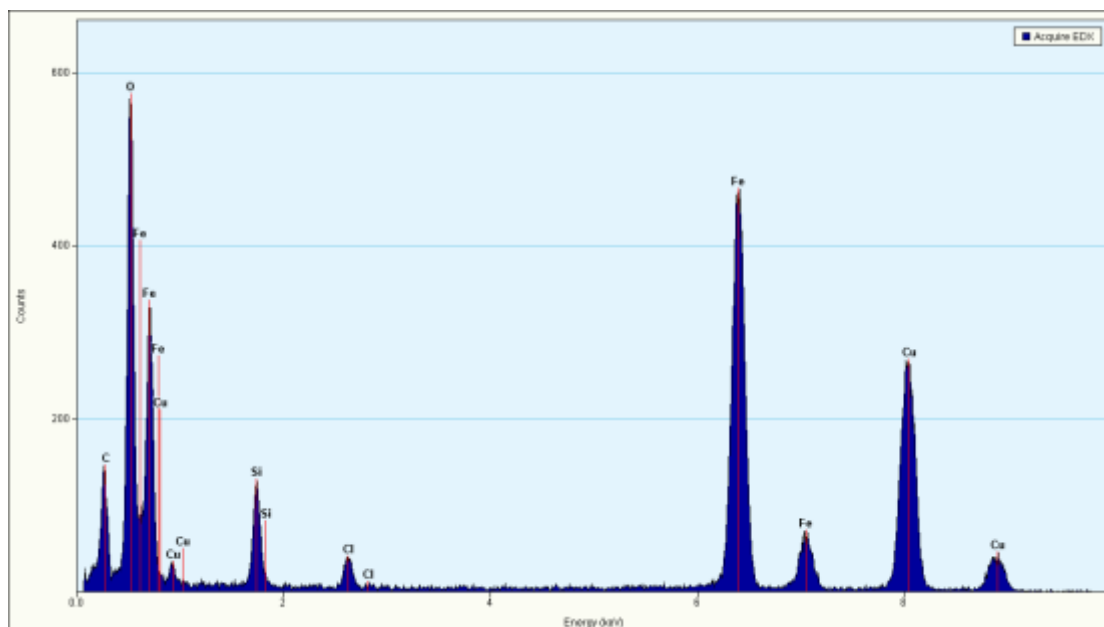


Figure S6 EDX pattern of the D SiO₂@Fe₃O₄ core/shell nanorod array/G architecture

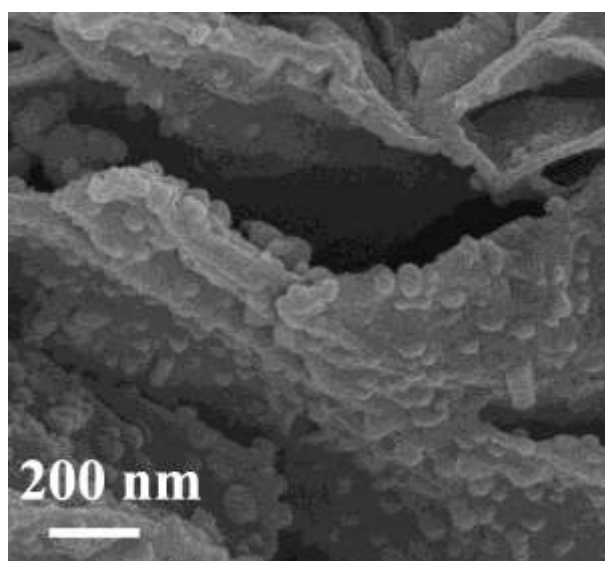


Figure S7 SEM image of the product without SiO₂ coating

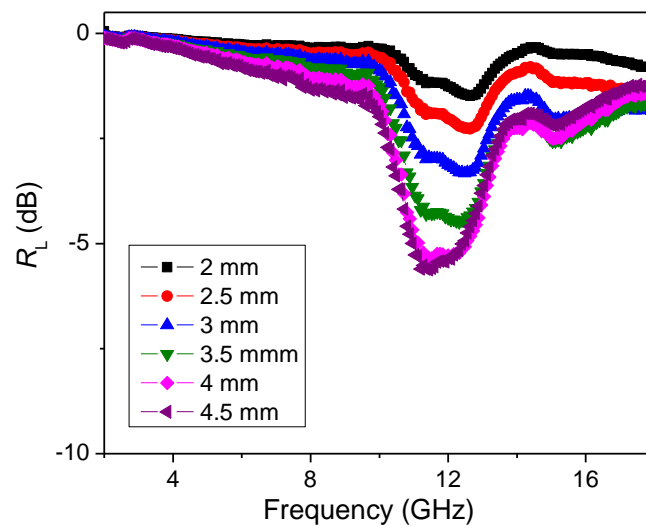


Figure S8 The reflection losses of the pure Fe_3O_4 nanorods

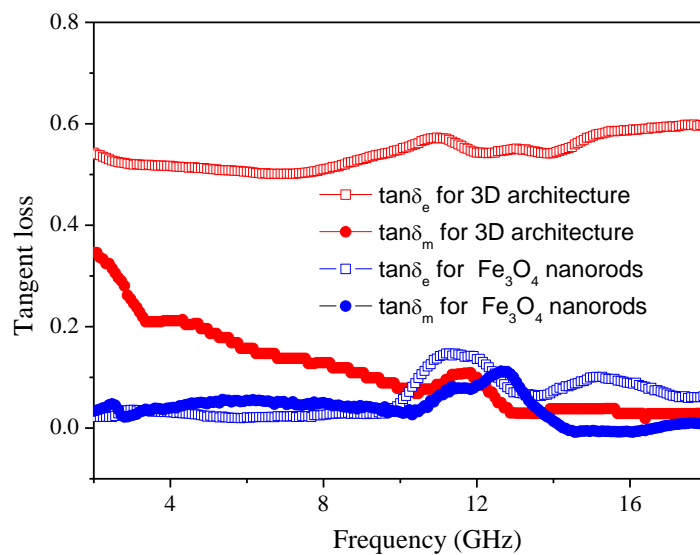


Figure S9 The tangent losses for 3D architecture and the pure Fe_3O_4 nanorods

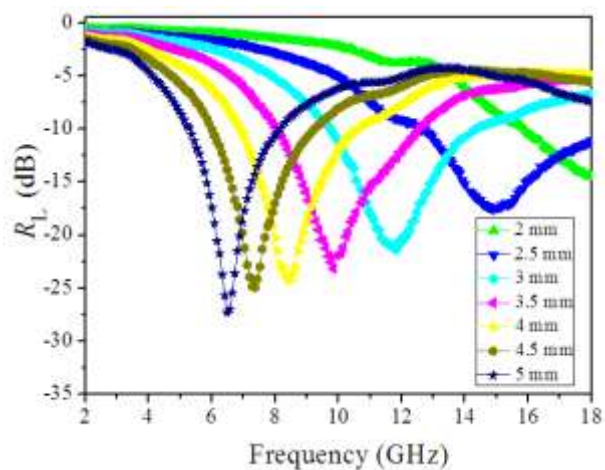


Figure S10 The reflection losses of 3D architecture with low loading of Fe_3O_4 nanorods

Notes and references

S1. W. S. Hummers and R. E. Offeman, *J. Am. Chem. Soc.*, 1958, **80**, 1339.