Supplementary Material to the article "Nanostructured GD_2O_3:YB micropowders for antibacterial hyperthermia"

S1. Synthesis procedure

Gadolinium nitrate hexahydrate, ytterbium nitrate pentahydrate, polyvinylpyrrolidone (PVP) and distilled deionized water were used as precursors for the synthesis. The polymer-salt method was used to synthesize the Gd_2O_3:Yb nanocrystalline powders. The synthesis route was as follows. The amounts of salts calculated according to the stoichiometric ratio and PVP were dissolved in distilled deionized water under intense stirring using the magnetic stirrer until the full dissolution of the chemicals. The solution was then dried in a drying chamber at a temperature of 70 °C. After that, obtained uniform organic-inorganic composition was calcinated in an electric muffle furnace at the temperature of 1000 °C during 2 hours. For a comparative study, the micropowder of Gd_2O_3 without ytterbium ions was prepared (sample #4). Samples #2 and #3 were characterized with a higher concentration of PVP in the stock solution comparing with sample #1. Sample #3 was additionally treated at 1000 °C for six hours. The chemical composition of the stock solutions and the calculated composition of the synthesized powders are given in the Table S1.

Table S1. Composition of stock solutions and calculated composition of the synthesized nanostructured micropowders

Sample	Chemical composition of stock solutions, mass. %				Synthesis and post-treatment conditions	Calculated chemical composition of synthesized nanostructured micropowders, mass. %	
	$Gd(NO_3)_3$	$Yb(NO_3)_3$	PVP	H_2O		Gd_2O_3	Yb
1	0.56	0.05	4.73	94.66	1000 °C/2 hours	98	2
2	0.55	0.05	6.93	92.47	1000 °C/2 hours	98	2
3	0.55	0.05	6.93	92.47	1000 °C/2 hours +1000 °C/6 hours	98	2
4	0.56	0	4.73	94.71	1000 °C/2 hours	100	0

S1. Material characterization

S1.1 SEM images

SEM images of the micropowders studied were obtained using a Phenom scanning electron microscope (FEI). Figure S1 shows examples of SEM images of the synthesized micropowders. It can be seen that the obtained samples consist of flat microflakes aggregates, characterized by a wide size distribution (Figure 1 a, c, e). On the other hand, Figure S1 shows that the above mentioned aggregates are composed of well resolved nanocrystallites (Figure 1 b, d, f).

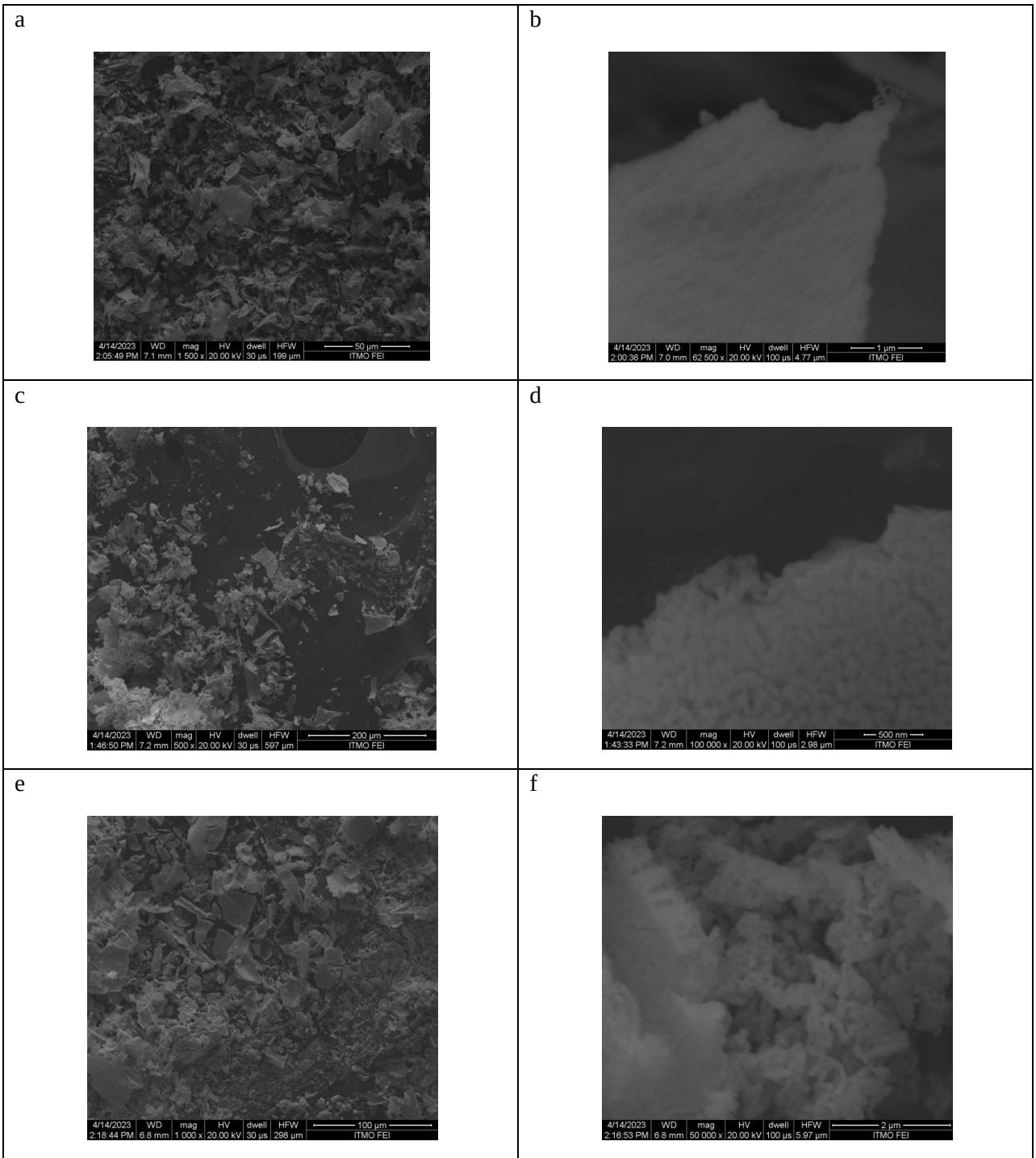

Figure S1. SEM images of micropowders Sample 1 – (a,b), Sample 2 – (c,d), Sample 3 – (e,f)

S1.2 Chemical composition

The chemical composition of the synthesized mircopowders was determined by X-ray fluorescence spectroscopy (ARL PERFORM'X, Thermo Scientific). The chemical composition was studied at room temperature under vacuum. For measurement, the powders were pressed into a tablet with boric acid. The recorded X-ray fluorescence signal was averaged over an area of the tablet (20 mm^2) with subsequent subtraction of the boric acid contribution. The experimental error was about 0.01 mol%. The chemical compositions of the synthesized powders determined by the X-ray fluorescence spectroscopy method are given in Table S2. The obtained values of ytterbium concentration are in good agreement with the calculated values given in Table S1.

Table S2. Chemical composition of Gd_2O_3:Yb powders obtained by X-ray fluorescence spectroscopy

Sample	Gd_2O_3, mass. %	Yb, mass. %
1	97,95	2,05
2	97,96	2,04
3	98,03	1,97

S1.3 XRD investigation

The average size of the nanocrystals in the micropowder as well as the lattice constant of the studied materials were estimated from the X-ray diffraction patterns recorded with an X-ray diffractometer (DRON-8, Burevestnik). The obtained XRD patterns are shown in Figure S2. All peaks in Figure S2 correspond to the crystalline phase of Gd_2O_3. The ratio of peak intensities is close to the standard (JCPDS 12-0797) [1]. The pattern does not reveal the presence of either amorphous phase or other crystalline phases.

The value of the lattice constant for cubic syngony can be estimated using the following expression

$$\frac{1}{d^2} = \frac{H^2+K^2+L^2}{a^2},$$

where *d* is spacing of lattice planes, calculated from the Wulff-Bragg equation based on the maximum of the corresponding peak in the diffraction pattern; *H*, *K* и *L* are Miller indices; a is the lattice constant [2]. The lattice constant values of the micropowders studied are given in the table S3. Each of the lattice constants in Table S3 is obtained by averaging the values calculated for different peaks.

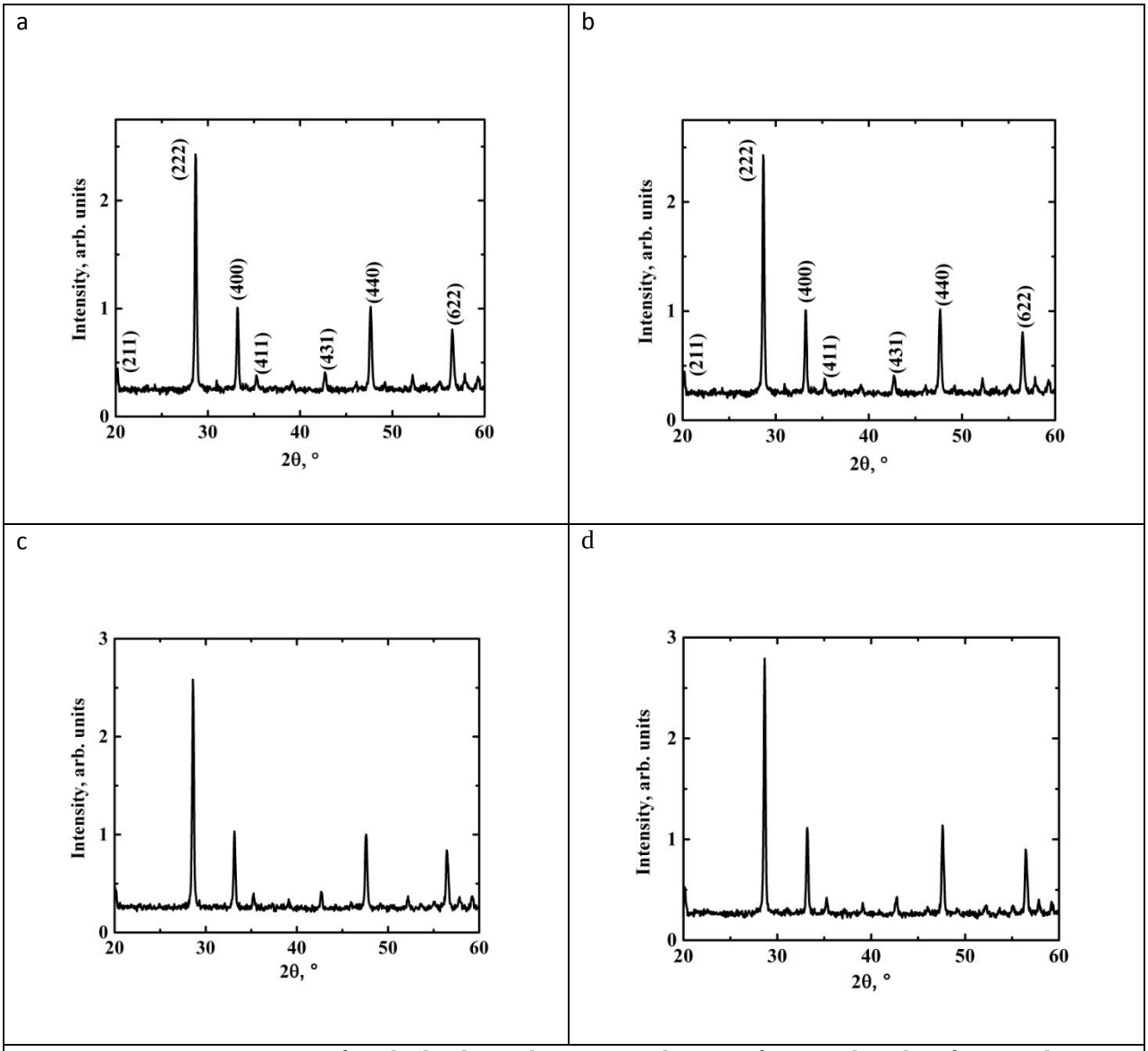

Figure S2. XRD patterns of studied Gd_2O_3:Yb micropowders, a is for Sample 1, b is for Sample 2, c is for Sample 3, d is for Sample 4

The average sizes *S* of the nanocrystals forming the micropowders were determined using the Scherrer formula [3]:

$$S = \frac{k\lambda}{\beta cos\theta},$$

where k is Scherrer constant (k=0.9 for a cubic crystal lattice), λ - X-ray wavelength, β - the band width of the most intense peak in an X-ray diffraction pattern, θ - Bragg angle. The estimated value of average nanocrystal sizes for the studied micropowders is shown in Table S3.

Table S3. Average size of nanocrystals and lattice constant of the studied nanostructured micropowders Gd_2O_3:Yb

Sample	Mean value of nanocrystals size, nm	Lattice constant, Å
1	38	10.791
2	35.5	10.783
3	32.9	10.787
4	34.6	10.797
Standart [4]	-	10.81

From the obtained data we can conclude, that increasing the concentration of PVP in the stock solution leads to a slight decrease in the average size of the nanocrystals. A decrease in crystal size with increasing PVP content was also previously shown in [5].

The lattice constant of control sample 4 without ytterbium ions is lower than the standard value (Table S3). The observation may be explained with the high specific surface area of nanoparticles. Doping of Gd_2O_3 material with ytterbium ions leads to a further reduces the lattice constant due to the difference in ionic radii of ytterbium (85.8 pm) [6] and gadolinium (93.8 pm) [7]. The observed reduction of the lattice constant confirms the incorporation of ytterbium ions into the structure of gadolinium oxide.

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