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The influence of a demagnetizing field on hysteresis losses in a dense assembly of superparamagnetic nanoparticles

S.A. Gudoshnikov [a,b,*], B.Ya. Liubimov [a], A.V. Popova [c], N.A. Usov [a,b]

[a] Pushkov Institute of Terrestrial Magnetism, Ionosphere and Radio Wave Propagation, Russian Academy of Sciences, (IZMIRAN), 142190, Troitsk, Moscow region, Russia
[b] Magnetic and Cryoelectronic Systems Ltd., 142190, Troitsk, Moscow region, Russia
[c] Institute on Laser and Information Technologies, Russian Academy of Sciences, (ILIT RAS), 142190, Troitsk, Moscow region, Russia

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ABSTRACT

The electrodynamic method is used to measure the hysteresis losses of a dense assembly of magnetite nanoparticles with an average diameter $D=25$ nm in the frequency range $f=10$–150 kHz and for magnetic field amplitudes $H_0=100$–300 Oe. It is found that the specific loss power is determined by a demagnetizing factor of a whole sample. It diminishes approximately 4.5 times when the sample aspect ratio decreases from $L/d=11.4$ to $L/d\approx 1$, where L and d are the sample length and diameter, respectively. For $H_0\leq 300$ Oe the maximal specific loss power 120 W/g is obtained for the sample with $L/d=11.4$ at $f=120$ kHz. For comparison, the assembly specific absorption rate has been determined also by means of direct measurement of the temperature difference between the inner and outer surfaces of a flat cuvette containing magnetic nanoparticles. For both methods of measurement close values for the specific absorption rate are obtained for samples with similar demagnetizing factors.

1. Introduction

The ability of superparamagnetic nanoparticles to absorb an energy of alternating external magnetic field can be used in magnetic nanoparticle hyperthermia [1,2], as well as for other applications, such as remote sintering of a mixture of magnetic nanoparticles with a biocompatible polymer powder. A number of experimental and theoretical investigations [3–17] have been carried out recently to determine the optimal sizes and material parameters of magnetic nanoparticles that provide sufficiently high specific absorption rate (SAR). Very high SAR values, of the order of 1 kW/g, have been reported for bacterial magnetosomes [7], for metallic cobalt nanoparticles [9], and for metallic iron nanocubes [14].

However, it has been pointed out [18] that the independent measurements on similar samples often provide significantly different SAR values. It was shown theoretically [15,16], that for a given set of the magnetic parameters, the SAR of a dilute assembly depends considerably on the nanoparticle size distribution. This fact may partly explain the difference in the SAR values reported by various authors for assemblies of similar chemical compositions. On the other hand, the experimental measurements have been carried out often on dense assemblies, where the influence of mutual interparticle interactions is appreciable. Here we prove experimentally that the hysteresis losses of a dense nanoparticle assembly depend significantly on the assembly demagnetizing factor.

The electrodynamic method [19] is used in the present paper to estimate the specific loss power (SLP) of a dense assembly of magnetic nanoparticles. This approach enables one to study the assembly hysteresis loop as a function of the frequency and the field strength. The shape and area of the low frequency hysteresis loop shows explicitly whether the properties of the assembly under investigation are optimal at a given frequency and magnetic field amplitude. Due to high sensitivity of the method small amounts (1–5 mg) of the magnetic nanoparticles are sufficient for the investigation. The latter is hardly possible for usual calorimetric measurement [10,11,14].

The low frequency hysteresis loops are obtained by means of integration of the electro-motive force (EMF) signal arising in a pick-up coil wrapped around a sample containing magnetic nanoparticles. The assembly SLP is known to be proportional to the area of the low frequency hysteresis loop [15,16], (see Eq. (2) below). The hysteresis loop measurements are carried out for magnetite nanoparticles with an averaged diameter $D=25$ nm in the frequency range $f=10$–150 kHz and for magnetic field amplitudes $H_0=100$–300 Oe. The results obtained demonstrate directly the dependence of the assembly hysteresis losses on the average

* Corresponding author at: Pushkov Institute of Terrestrial Magnetism, Ionosphere and Radio Wave Propagation, Russian Academy of Sciences, (IZMIRAN), 142190, Troitsk, Moscow region, Russia. Tel.: +7 496 751 97 46; fax: +7 496 751 97 68.
E-mail address: gudosh@izmiran.ru (S.A. Gudoshnikov).

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demagnetizing factor of the whole sample. It is found that the SLP of the assembly diminishes approximately 4.5 times when the sample aspect ratio decreases from $L/d=11.4$ to $L/d\approx 1$, where L and d are the sample length and diameter, respectively. Evidently, for a dense assembly of magnetic nanoparticles the average demagnetizing field reduces significantly the externally applied alternating magnetic field.

To confirm the results of the hysteresis loss measurement we carried out also heating power measurements to determine the SAR of the same nanoparticle assembly directly. (Note that the term SAR will be used below only for direct heating power measurements.) In the experiment performed we measured the thermal difference that arises between the inner and outer surfaces of a flat cuvette containing the magnetic nanoparticles in the alternating magnetic field. We would like to note that the usual optical measurement [8] of a sample surface temperature leads to a considerable uncertainty in the volume energy release evaluation, because the surface temperature depends on the heat exchange conditions at the sample surface. On the other hand, a simple theoretical estimate shows that a steady thermal gradient within the wall of a flat cuvette does not depend on the unknown heat exchange conditions in the ambient media. Therefore, the measurement of the thermal gradient provides a reliable estimate for a volume energy release within the cuvette. It is shown that the hysteresis loss and thermal measurements lead to close values for samples with similar demagnetizing factors. This ensures the reliability of both of the measurements used. The dependence of a SAR or SLP value on the sample demagnetizing factor has to be taken into account when one compares the specific absorption rates reported by different authors.

The results obtained seem important not only for application in magnetic nanoparticle hyperthermia, but also for remote sintering of a mixture of a low-melting plastic material with magnetic nanoparticles. It is shown that only ten weight percents of the magnetic nanoparticles in a mixture with a polylactide powder is sufficient to provide fast sintering of a sample of the order of 1 cm in size. The composite material obtained shows mechanical characteristics suitable for tissue engineering.

2. Experimental

2.1. EMF measurements

To measure the low frequency hysteresis loops of magnetic powder samples we employed electrodynamic method [19] that is routinely used for investigation of the frequency behavior of ferromagnetic materials. Our installation consists of a home-made frequency-adjustable electromagnet designed similar to [20] and a recorder of the EMF signals that arise in the pick-up coils in the alternating external magnetic field. The alternating magnetic field of sufficient amplitude is created using the generator of sinusoidal signals, the power amplifier Parasaund-A21, and the series resonance circuit comprised of the capacitance bridge and the inductor having EPCOS ferrite core.

To study the frequency dependence of the assembly hysteresis losses, the frequency adjustment of the capacitance loop of the source of the alternating magnetic field can be used. The maximal value of the alternating magnetic field amplitude achieved for the installation is given by $H_0=600$ Oe at a frequency $f=20$ kHz. However, it diminishes up to $H_0=200$ Oe when the frequency increases till $f=200$ kHz. In this work most of the measurements were done in the range of frequencies $f=10$–150 kHz and field strengths $H_0=100$–300 Oe.

Two pick-up coils (95 turns of diameter 0.15 cm) with opposite connection are used to measure the EMF signal generated by a sample containing magnetic nanoparticles. In the absence of the magnetic nanoparticles the non-compensated signal arising in the alternating external magnetic field does not exceed 1 mV. However, the EMF signal increases proportionally to the derivative of the sample total magnetic moment when the sample is inserted in one of the pick-up coils. Another EMF signal arises in the field pick-up coil that contains 10 turns of diameter 0.33 cm. It is used to measure the amplitude H_0 of the alternating external magnetic field.

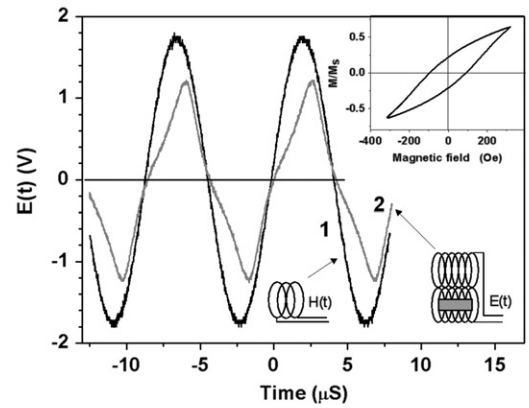

Fig. 1. EMF signals generated in the applied alternating magnetic field in the field coil (1) and in the pick-up coil (2) containing the magnetic nanoparticles. The inset shows the assembly hysteresis loop corresponding to the EMF signal (2).

Fig. 1 shows EMF signals arising in the corresponding pick-up coils at a frequency $f=112$ kHz and magnetic field amplitude $H_0=280$ Oe for the sample with aspect ratio $L/d=5.9$. In spite of a small amount of the magnetic material (1.4 mg) used for the sample preparation one can see that the amplitude of the EMF signal of magnetic nanoparticles reaches a value of the order of 1 V. The EMF signals of similar amplitude are generated in the range of frequencies 70–100 kHz and field strengths 200–300 Oe. Using the Maxwell equations it is easy to see that the total magnetic moment of a whole sample can be calculated as a function of the time by means of the relation:

$$<MV> = \frac{cL_c}{4\pi N_c}\int E(t)dt, \qquad (1)$$

where c is the light velocity, N_c/L_c is the number of turns per unit length for the pick-up coil and $E(t)$ is the EMF signal. Eq. (1) can be justified assuming the axial symmetry of the magnetization distribution within the sample and supposing that it is placed in a sufficiently long coil. The upper inset in Fig. 1 shows the low frequency hysteresis loop of the assembly that is obtained by means of integration of the EMF signal 2 according to the Eq. (1).

Integrating numerically the hysteresis loop area in the variables MV–H, multiplying the result by a frequency of the alternating magnetic field and dividing it by a mass Q_m of the magnetic content of the powder in grams one obtains the SLP of the assembly as

$$SLP = 10^{-7}\frac{f}{Q_m}\oint <MV> dH\ (W/g). \qquad (2)$$

2.2. Thermal measurements

To determine directly the SAR of an assembly in the alternating external magnetic field and to compare the data with the hysteresis loss measurements, the volume energy release has been measured in a flat cylindrical cuvette made of Teflon (see inset in Fig. 2). The cuvette has an outer diameter 2.8 cm

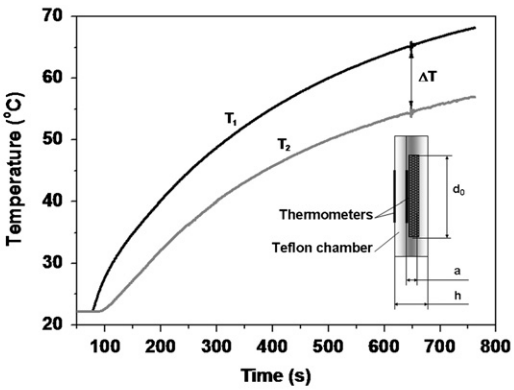

Fig. 2. The temperatures at the inner (T_1) and outer (T_2) surfaces of a flat cylindrical cuvette as a function of time during the heating of the inner cavity by a calibrated electrical resistor. The dissipated power is given by 1.1 W.

and a height $h=0.8$ cm. The inner cylindrical cavity within the cuvette, where the magnetic nanoparticles can be placed, has a diameter $d_0=2.0$ cm and a height $a=0.2$ cm, respectively. Therefore, the thickness of the heat insulating layer along the cuvette axis is given by $(h-a)/2=0.3$ cm. Thin copper thermometers have been fastened at the inner and outer surfaces of the cuvette to measure the temperature difference arising during the sample heating.

Taking into account nearly flat geometry of the cuvette it is easy to see that a steady thermal gradient sets up within the heat insulating layer when the transient thermal process is finished. The steady difference $\Delta T=T_2-T_1$ between the inner and outer cuvette surfaces is given by

$$\Delta T=\frac{U_0}{4k_t}a(h-a) \tag{3}$$

Here U_0 is the intensity of the volume energy release within the cavity and k_t is the thermal conductivity of the heat insulating layer. It is clear from the energy conservation law that in the stationary regime the heat generated per unit time within the Teflon cuvette is to flow through the Teflon walls. As a result, the thermal difference (3) does not depend on the heat exchange conditions at the outer cuvette surface, in contrast to the temperatures T_2 and T_1 themselves. It does not depend also on the temperature distribution within the flat cuvette along its axis.

The thermal conductivity k_t can be measured separately generating the known thermal power within the cavity and measuring the corresponding thermal difference ΔT. Fig. 2 shows the temperatures of the inner T_1 and outer T_2 cuvette surfaces as the functions of the time during a uniform heating of the inner cavity by means of the calibrate electrical resistor at a power $P=1.1$ W. Using these data the layer thermal conductivity has been estimated to be $k_t\approx 4.7\times 10^4$ erg/(cm × s × grad). Then, the specific energy release within the cavity at a given frequency and magnetic field amplitude can be calculated as

$$SAR=\frac{VU_0}{Q_m}=10^{-7}\frac{\pi d_0^2 k_t \Delta T}{(h-a)Q_m}\ (W/g), \tag{4}$$

where ΔT is the temperature difference (3) and $V=\pi d_0^2 a/4$ is the inner cavity volume.

3. Results and discussion

Magnetite nanoparticles were procured from Nanostructured & Amorphous Materials, Inc. [21]. The magnetite powder density was around 1 g/cm^3. The particles had nearly spherical shape and can be characterized by a log-normal size distribution with an average diameter $D=25$ nm and a dispersion $\sigma=0.3$. The powder x-ray diffraction confirmed that magnetite was the main crystalline phase. A quasistatic hysteresis loop of the assembly was measured at a room temperature using vibration magnetometer VM-2K. The assembly coercive force is given by $H_c=50$ Oe. The maximum magnetization at applied magnetic field $H=5$ kOe is determined to be $M(5\text{ kOe})=43$ emu/g. The reduced value of the high field magnetization with respect to saturation magnetization of bulk magnetite, $M_s=80$ emu/g, is attributed to the presence of protected nonmagnetic layers at the particle surfaces. Based on this assumption, the actual magnetic content of the powder was estimated as $Q_m=43Q_s/80$, where Q_s is the mass of the powder used for a sample preparation.

3.1. Hysteresis loss measurement

Thin plastic tubes of various inner diameters, $d=0.07$–0.14 cm, were used to prepare the samples for the hysteresis loss measurement. The tubes were filled up with small amounts (1–5 mg) of dry magnetite powder of the same density 1 g/cm^3. After tube filling their ends were closed with thin membranes. For every sample the true mass of the powder was carefully measured. The cylindrical samples of different length, $L=0.15$–0.8 cm, and aspect ratios, $L/d=1$–11.4, were used in the experiment to study the dependence of the SLP of a dense nanoparticle assembly on the sample demagnetizing factor. The total magnetic moment of a sample as a function of time can be obtained integrating the EMF signal of magnetic nanoparticles according to Eq. (1). Then the SLP of the assembly under investigation can be calculated by means of Eq. (2).

Fig. 3 shows the SLP of the sample with aspect ratio $L/d=5.9$ as a function of H_0 for various alternating magnetic field frequencies. One can see that for magnetic field amplitudes $H_0<250$–280 Oe there is a linear regime [15,16], when the assembly magnetization is proportional to H_0, whereas the SLP of the assembly is proportional to its square, SLP$\sim H_0^2$. However, in agreement with the numerical simulation data [16], a non-linear regime realizes for high enough magnetic field amplitudes, $H_0>300$ Oe. In this case the hysteresis losses are nearly proportional to H_0. As a rule, the nonlinear regime is necessary to heat a sample to a high temperature $T\sim 100$ °C.

Making SLP measurements for the samples with various aspect ratios we confirmed that the average demagnetizing field influences considerably on the energy absorption process in a dense assembly of magnetic nanoparticles. Fig. 4 shows the SLP of the samples with various aspect ratios as a function of the alternating magnetic field frequency. It is evident from Fig. 4, that the highest

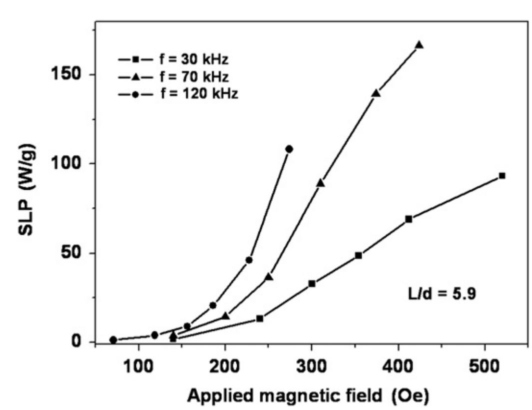

Fig. 3. Specific loss power of the sample with aspect ratio $L/d=5.9$ as a function of the alternating magnetic field amplitude at various frequencies.

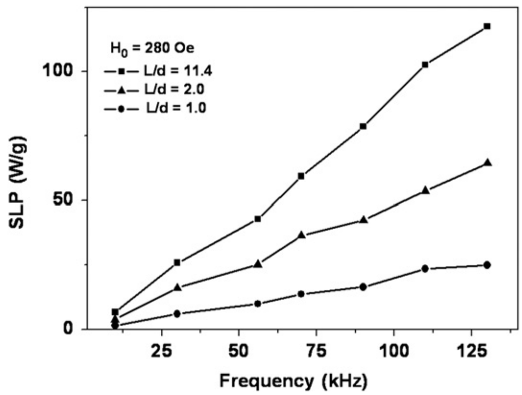

Fig. 4. Specific loss power as a function of frequency for Fe_3O_4 nanoparticle samples with various aspect ratios L/d.

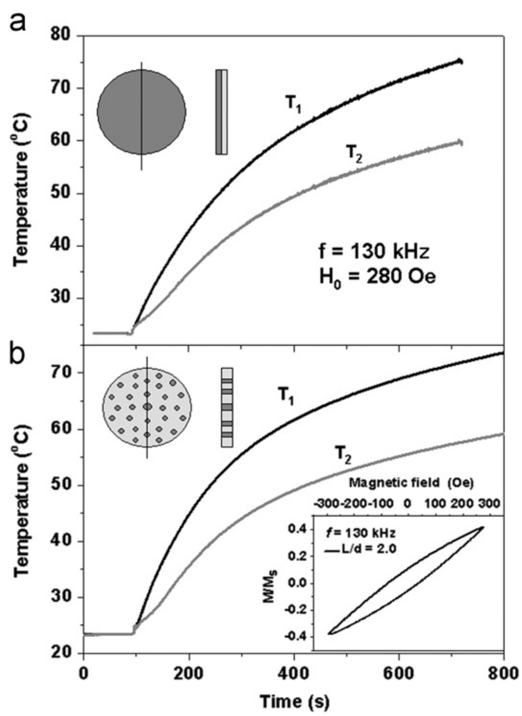

Fig. 5. (a) The temperatures at the inner (T_1) and outer (T_2) surfaces of a flat cylindrical cuvette as a function of time for the uniform sample #1 with a small aspect ratio 0.05. (b) The same data for the perforated sample #2 consisting of 28 small cylinders with aspect ratio 2.0. The insets show schematically the geometry of the samples.

SLP value is obtained for the sample with the largest aspect ratio, $L/d=11.4$, that has the lowest demagnetizing factor, $N\approx 0$. In this case the average demagnetizing field within the sample is small. Therefore, it is only slightly decreases the applied alternating magnetic field acting on the magnetic nanoparticles.

3.2. Thermal measurements

The alternative SAR measurements were carried out using the method described in Section 2.2. Three different samples were prepared for this purpose. In the sample #1 the magnetic nanoparticles were placed in a cylindrical cavity of a flat Teflon cuvette with the inner diameter $d_0=2$ cm. The thickness of the layer of the magnetic nanoparticles was $a_1=0.1$ cm. The remainder part of the cavity volume was filled up by a substance with a high thermal conductivity (see inset in Fig. 5(a). Note that the total mass of the powder used to prepare this sample, $Q_s=420$ mg, is approximately 100 times larger than the typical mass of the nanoparticles used for the hysteresis loss measurements. When the cuvette was placed in the alternating magnetic field with the frequency $f=130$ kHz and magnetic field amplitude $H_0=280$ Oe, the temperatures T_1 и T_2 at the inner and outer surfaces of the cuvette were recorded as a function of time, as shown in Fig. 5(a). Using the steady temperature difference $\Delta T=T_2-T_1$ obtained in this experiment, the SAR of the assembly was calculated by means of Eq. (4) to be 6.5 W/g. So small SAR value in comparison with the hysteresis loss measurements is explained by a large demagnetizing factor of a flat sample, $N\approx 4\pi$, as the layer of the magnetic nanoparticles for the sample #1 has very small aspect ratio, $a_1/d_0=0.05$.

To compare directly the results of the thermal and hysteresis loss measurements we prepared the perforated sample #2 as Teflon disk of diameter $d_0=2$ cm having 28 holes uniformly distributed over its area (see upper inset in Fig. 5(b). The holes have the height 0.2 cm and diameter 0.1 cm. They were uniformly filled up by the magnetite powder, the total mass of the nanoparticles for the perforated sample being $Q_s=56$ mg. Therefore, the sample #2 consists of widely spaced cylinders with the aspect ratio $L/d=2.0$. The disk with magnetic nanoparticles was placed in the internal cavity of the cuvette. The results of the thermal measurement for the sample #2 at the same frequency, $f=130$ kHz, and field strength, $H_0=280$ Oe, are shown in Fig. 5b. One can see that the perforated sample is heated with nearly the same rate as that of sample #1, in spite of the fact that it contains 8 times smaller mass of the magnetic nanoparticles with respect to the sample #1. The SAR of the perforated sample was estimated to be 24.2 W/g. On the other hand, the electrodynamics measurement for a control sample with the diameter $d=0.1$ cm and length $L=0.2$ cm at the same frequency and magnetic field amplitude resulted in the SAR value 25.3 W/g. The low frequency hysteresis loop for the control sample is shown in the lower inset in Fig. 5(b). It is proved therefore, that for samples with close demagnetizing factors the hysteresis loss and thermal measurements lead to nearly the same SAR values.

Fig. 6 shows the inner temperature T_1 recorded for the uniform sample #3 having the diameter 1.5 cm, height 0.2 cm, and the aspect ratio 0.13. For this sample at similar heating conditions ($f=130$ kHz, $H_0=280$ Oe) the maximal temperature achieved within the cavity exceeds 150 °C. It is twice larger than the cavity temperature obtained for the sample #1 at the end of the treatment (see Fig. 5(a), curve T_1). Note that the total mass of the nanoparticles in the sample #3, $Q_s=430$ mg, is close to that of sample #1. Therefore, the thermal measurements demonstrate

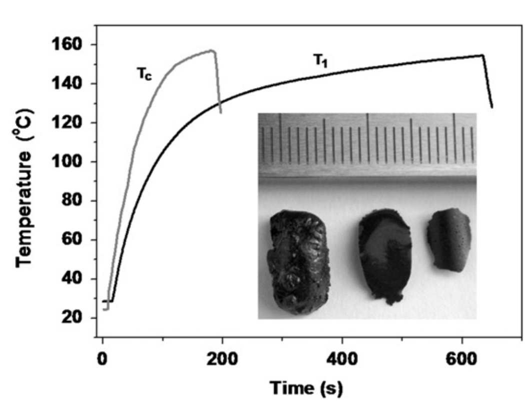

Fig. 6. The curve labeled T_1 shows the time dependent cavity temperature recorded for the uniform sample #3 with the aspect ratio 0.13. The curve labeled T_c shows surface temperature of a composite sample that is a mixture of 90 wt% of polylactide powder and 10 wt% of magnetic nanoparticles. The inset shows the examples of sintered composite samples.

explicitly the dependence of the SAR of a dense nanoparticle assembly on the sample demagnetizing factor.

In addition, a complete sintering in the alternating external magnetic field a mixture of polylactide particles with a small amount of magnetite nanoparticles has been demonstrated. Polylactide (PDL-04, Purac Biomaterials, Netherlands) is a bio-compatible polymer of a polylactic acid, which is widely used in tissue engineering. The polylactide particles have an average diameter ~80 μm. To prepare the mixture we used 90 wt% of polylactide (white powder) and 10 wt% of magnetic nanoparticles (black powder). The components were thoroughly mixed until a homogeneous gray powder was obtained. The uniformity of the mixture was controlled by means of an optical microscope. As Fig. 6 shows (curve labeled T_c), under the influence of the alternating magnetic field (f=130 kHz, H_0=280 Oe) the surface temperature of a composite sample increases up to 150 C during 200 s. The surface temperature of the sample was measured by means of the infrared imager IRTIS-2000. It is found that the melting of the composite samples occur above T=60 C. After melt freezing a uniform solid material is formed. The inset in Fig. 6 shows the photo of the sintered polylactide samples.

4. Discussion and conclusions

Superparamagnetic nanoparticles possess a unique capability to absorb the energy of a low frequency alternating magnetic field of moderate amplitude. Due to small particle sizes the thermally assisted transitions between magnetic potential wells become possible even at room temperature and for magnetic field amplitudes significantly lower than the characteristic particle anisotropy field. This leads to a magnetic hysteresis and to appreciable specific absorption rate for a superparamagnetic nanoparticle assembly in alternating external magnetic field. It is well known [15,16] that the SAR of the assembly depends on the particle size distribution and their magnetic parameters. In the present paper we draw attention to the fact that the SAR of a dense nanoparticle assembly is determined also by a demagnetizing factor of a whole sample.

In the given paper the electrodynamic method is applied to measure the low frequency hysteresis loops of a nanoparticle assembly. The installation developed is capable to measure assembly hysteresis loops in the range of frequencies 10–150 kHz, at alternating magnetic field amplitudes up to 300 Oe. It is shown that SLP of a dense nanoparticle assembly is determined by a demagnetizing factor of a whole sample. Actually, the SLP diminishes approximately 4.5 times when the aspect ratio of a dense nanoparticle assembly decreases from L/d=11.4 to $L/d \approx 1$. This fact has to be taken into account when one compares the results of hysteresis loss measurements on similar nanoparticle assemblies. For a dense assembly of Fe_3O_4 nanoparticles with average diameter D=25 nm a maximal SLP in moderate magnetic fields $H_0 \leq 300$ Oe is obtained to be 120 W/g for a sufficiently elongated sample with aspect ratio L/d=11.4 at a frequency f=120 kHz. It is demonstrated also that a complete sintering of a low-melting polylactide powder containing only 10% of the magnetic nanoparticles can be achieved after application of the alternating magnetic field during 200 s.

Acknowledgments

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