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Design of an In-Situ Magnetic Particle Spectrometer (INSPECT II) for co-precipitation methodology-based synthesis

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Abstract

Magnetic particle spectrometry (MPS) is a characterization tool, which helps in determining the magnetic properties of Magnetic nanoparticles (MNPs). MNPs heavily influence image resolution, hence intensive research is conducted to improve the magnetic properties of these MNPs. In-situ magnetic particle spectrometer (INSPECT) was first presented in 2019 and has the capability to track the nucleation and growth of MNPs. The presented research discusses the design and development of the second generation of INSPECT which investigates the impact of a fully controllable temperature bath, lower power requirements, and a new hybrid housing design for carrying out synthesis at higher temperatures. This research paper deals with the development of different hardware modules and sensitivity profiles of INSPECT II.

I. Introduction

In the last few decades, magnetic nanoparticles (MNPs) have been utilized in various fields of technology and life sciences. Particularly, in the field of biotechnology and medicine, nanoparticles prove beneficial. For instance, in medicine nanoparticles can be used as carriers to administer chemotherapeutic drugs to treat tumors furthermore, magnetic nanoparticles can be used for thermo-therapeutic cancer treatment. In biotechnology, magnetic properties are used for cell separation and manipulation techniques. Magnetic particle imaging (MPI) is a new medical imaging technique that images the spatial distribution of these MNPs. Due to their high saturation magnetization and relaxation behavior and due to their biocompatibility, MNPs prove to be the perfect candidate as a tracer material for MPI imaging [1]. MNPs can be synthesized using many synthesis techniques, and the quality of the image for MPI depends on the characteristics of the synthesized nanoparticles [2]. Today, there are a number of devices to characterize the magnetic properties of MNPs, one of them is the magnetic particle spectrometer (MPS) another one is the vibrating sample magnetometer (VSM). Both of these devices can characterize the magnetic properties of the synthesized MNPs, but they are unfit to record the magnetic properties during the synthesis process itself. Although there are techniques that provide real-time data about the nucleation and growth of MNPs such as real-time transmission electron microscopy and X-ray scattering, these devices require special chambers for synthesis and a good amount of user expertise. Additionally, these devices are also quite expensive. This research focuses on the design and development of a In-situ magnetic parti-

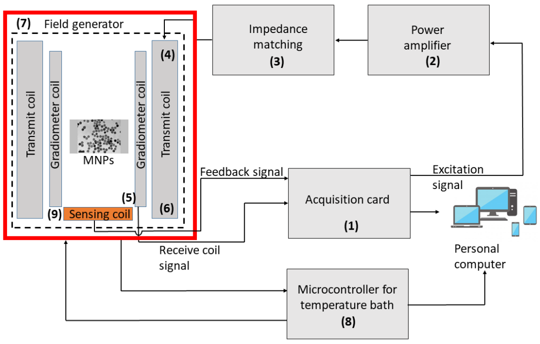

Figure 1: Block diagram consisting of the different modules of INSPECT

cle spectrometer (INSPECT II) with a fully controllable temperature bath for maximizing control of the synthesis environment and simultaneously determining the magnetic properties of MNPs during the synthesis process [3]. The device itself is autonomous, requires no user expertise and more importantly, the synthesis can be carried out in a 50 ml glass flask used in routine synthesis processes.

II. Methods and materials

Figure 1 shows the different modules of INSPECT II, which are (1) the acquisition card which is Red Pitaya producing the excitation signal of 25 kHz and also receiving the MNPs response. (2) The power amplifier amplifies the excitation signal from the acquisition card and sends it to (3) the impedance matching unit, which matches the reactance of the transmit coil to the desired impedance of the power amplifier. (4) The signal is then finally fed to the transmit coil in the field generator unit, which produces the magnetic field required to excite the MNPs. (5) The receiving side of the gradiometer coil acquires the MNPs response and sends it back to the acquisition card to be analyzed. To monitor the amount of field produced by the transmit coil (6), a sensing coil (9) is concentrically placed inside the transmit coil and this voltage signal is also fed back to the acquisition card. Furthermore, there is a dedicated (7) heating bath which could be controlled with the help of an (8) microcontroller to control the duration as well as the temperature of the synthesis. In the end, a personal computer is used for displaying, storing, and analyzing data. In the next subsections, an overview of different modules is presented.

II.I. Power amplifier

In order to reach the required magnetic field of 10 mT to excite the MNPs, the excitation signal needs to be amplified. This is achieved with the help of a power amplifier. The power amplifier used for INSPECT II is the AE Techron 2105 (AE Techron, USA), providing up to 1000 W at a maximum of 8 Ω.

II.II. Impedance matching

The impedance-matching unit transfers the maximum power from the power amplifier to the field generator, while reducing the amount of reflected power. INSPECT II uses capacitive impedance matching, in which there are two capacitors. Firstly, C_P is connected in parallel to the transmit coil and power amplifier. Secondly, C_S is connected in series to the transmit coil. The capacitance value of C_P was calculated to be 5.42 µF and the value of C_S was calculated to be 0.21 µF. The impedance was matched at 3.04 Ω at a phase of −4.35° at 25 kHz.

II.III. Transmit coil

The transmit coil has a diameter of 74 mm and a total height of 46.8 mm. To construct the transmit coil, a Litz wire (2000 x 0.05 mm) strains with a total diameter of 2.6 mm was used. The coil has a resistance of 195.67 mΩ and an inductance of 332.43 µH at 25 kHz. The total power consumption to produce a field of 10 mT is approximately 38 W with an input current of 14 A.

II.IV. Gradiometer coil and feedback

The gradiometer coil consists of a cancellation coil wound in the opposite direction to cancel out the excitation signal and a receive coil to capture the particle response. It is essential to remove the excitation signal, as the magnitude of the excitation signal is 5-10 times higher than the MNPs response [4]. The gradiometer coil was constructed using a copper wire with a diameter of 0.4 mm. The coil was wound on a hollow cylindrical housing with a diameter of 61 mm, where the receive coil comprises 20 turns and the cancellation coil comprises 25 turns. To generate the feedback signal a sensing coil ((9) in Figure 1) of copper wire with the same diameter is coiled around the upper edge of the gradiometer coil to estimate the field generated by the transmit coil.

II.V. Housing for the field generator

The housing for the field generator houses the transmit coil and the gradiometer coil is designed using SolidWorks 2020 (Dassault Systemes, France). The housing was later 3D printed using Form 3B (Formlabs Inc., USA). The material used for printing is a High-Temp Resin from Formlabs Inc., with a heat deflection temperature (HDT) of 238 °C at 0.45 MPa. To prevent excessive heating of the transmit coil, the housing contains small cooling channels which allow air to pass through to the surface of the coil and is connected to a pressurized air supply to the top of the housing. The 3D printed field generator with

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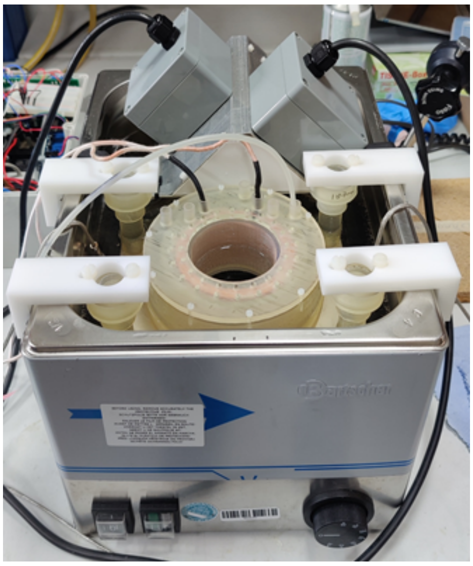

Figure 2: The field generator is placed inside the heating basin, which can be programmed to heat the contents inside up to user-defined temperature and duration.

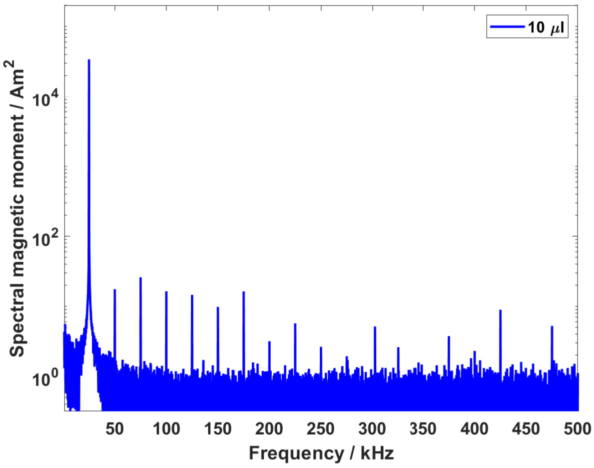

Figure 3: Measurements showing the sensitivity of INSPECT II using commercially available MNPs known as Resovist. The graphs show the amplitude spectrum of SPIONs for 10 µL.

the heating unit and its support structure is shown in Figure 2.

II.VI. Heating Bath

The new design of the heating bath consists of a stainless steel basin with a built-in heater and two additional immersion heaters (heating rods), which can be controlled with the help of a microcontroller to vary the heating-up time depending on the synthesis. The total volume in the heating bath is a maximum of seven liters which further reduced the heating time in comparison to the previous version of INSPECT [3]. The heating bath has a maximum output power of 3800 W. Furthermore, the design consists of a 12 V power supply for the microcontroller, power relays with a maximum output of 30 A, a measuring unit for Pt100 temperature sensors, and an LCD screen for displaying the temperature. Figure 2 shows the complete INSPECT II with the heating bath and the field generator.

III. Experiments and Results

Different experiments were conducted to check the efficiency of the heating bath and furthermore, the sensitivity of the field generator is determined with the help of commercially available MNPs. When using both heating rods in addition to the heating basin it took approximately 14.5 min for a temperature change from 1°C to 80°C for 6 liters of water. Moreover, the heating process can be extended by automatically switching the heating bath with the help of the software. To determine the sensitivity of INSPECT II, commercially available MNPs called Resovist (from Bayer, Germany) was used [5]. Resovist is a multicore particle with an iron oxide core, consisting of many single crystals, which have a diameter of approximately 4.2 nm. The test samples consisted of five different volumes namely 10, 20, 30, 40, and 50 µL respectively. Figure 3 shows the amplitude spectrum of the 10 µL sample. The odd harmonics could be easily detected till 375 kHz showing enough sensitivity to detect the early stages of nucleation.

IV. Conclusion

The research consists of designing and developing a newer version of INSPECT. The current research paper focused on the hardware development and testing of vital modules like the heating bath and the sensitivity of the field generator. In a time period of 14.5 min, a temperature change from 1°C to 80°C for 6 liters of water was observed with the new heating bath. Furthermore, the field generator was able to detect 10 µL samples of commercially available MNPs to 475 kHz which corresponds to 19 harmonics.

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Author's statement

Conflict of interest: Authors state no conflict of interest. Informed consent: Informed consent has been obtained from all individuals included in this study.

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