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Magnetic moment investigation by frequency mixing techniques

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Gas turbines and other large industrial equipments are subjected to high-temperature oxidation and corrosion. Research and development of efficient protective coatings is the main task in the field. Also, knowledge about the depletion state of the coating during the operation time is important. To date, practical nondestructive methods for the measurement of the depletion state do not exist. By integrating magnetic phases into the coating, the condition of the coating can be determined by measuring its magnetic properties. In this paper, a new technique using frequency mixing is proposed to investigate the thickness of the coatings based on their magnetic properties. A sensor system is designed and tested on specific magnetic coatings. New approaches are proposed to overcome the dependency of the measurement on the distance between coil and sample that all noncontact techniques face. The novelty is a low cost sensor with high sensibility and selectivity which can provide very high signal-to-noise ratios. Prospects and limitations are discussed for future use of the sensor in industrial applications.
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I. INTRODUCTION

In many industrial applications, metallic and nonmetallic Cr- and/or Al-rich protective coatings are applied to protect against high temperature corrosion, oxidation, or physical degradation.[1,2] After long operation periods, progressive oxide scale formation and interdiffusion between the coating and the substrate leads to the depletion of the scale-forming elements. Decaying of their concentration under a critical value results in accelerated corrosion and quick damage of the coating.[3] To date, several techniques for investigation of the depletion state of metallic layers are available, e.g., eddy current inspection.[4,5] For ceramics and other nonconductive protective coatings, only destructive methods are available.[6] By integration of magnetic phases into the coating and measuring the magnitude of their magnetic properties, important information about the coating's condition can be gained, e.g., thickness.

In this paper, a new technique based on frequency mixing is proposed to investigate the thickness of magnetic thin coatings. The thickness is indirectly determined by measuring magnetic properties of the coating. The performance of the method is investigated on defined magnetic samples with a special focus on eliminating cross-sensitivities due to the distance variations between sensor and sample.

II. FREQUENCY MIXING TECHNIQUE

Frequency mixing is a common technique in many fields: sensors,[7] spectroscopy,[8] electronics,[9] etc. In general, it is used to extract small signals from noisy backgrounds. The method has already been extended to measure the magnetic properties of particles and the detection of magnetic nanoparticles.[10,13]

Based on this approach, the magnetic material is exposed to a magnetic field consisting of two frequencies $f_1 \gg f_2$, as shown in Fig. 1(a). The amplitude of the low frequency component of the field is chosen to be high enough to ensure that the magnetic material is periodically in saturation. As a result, the response signal of the material [Fig. 1(c)] is a linear combination of both frequencies $(a \cdot f_1 + b \cdot f_2)$ [Fig. 1(e)]. The appearance of these components is highly specific to the nonlinear magnetization curve of the material [Fig. 1(b)]. The higher harmonics in the mixing signal [Fig. 1(e)] will be generated only if ferromagnetic materials are present. Other materials in the interaction volume such as ceramics, metals, etc., will not produce any frequency mixing signals if their magnetic behavior is linear, i.e., paramagnetic or diamagnetic in the considered range of the magnetic field. Therefore, by measuring the mixed signal [Fig. 1(c)], information about the ferromagnetic material content can be obtained.

Because of the good selectivity compared to other methods,[14] frequency mixing has become a standard technique for tracking and measuring magnetic markers in biological and medical applications.[10–13] Based on these developments, the method is adapted to investigate the magnetic properties and thickness of magnetic thin coatings, which will be described in the next chapter.

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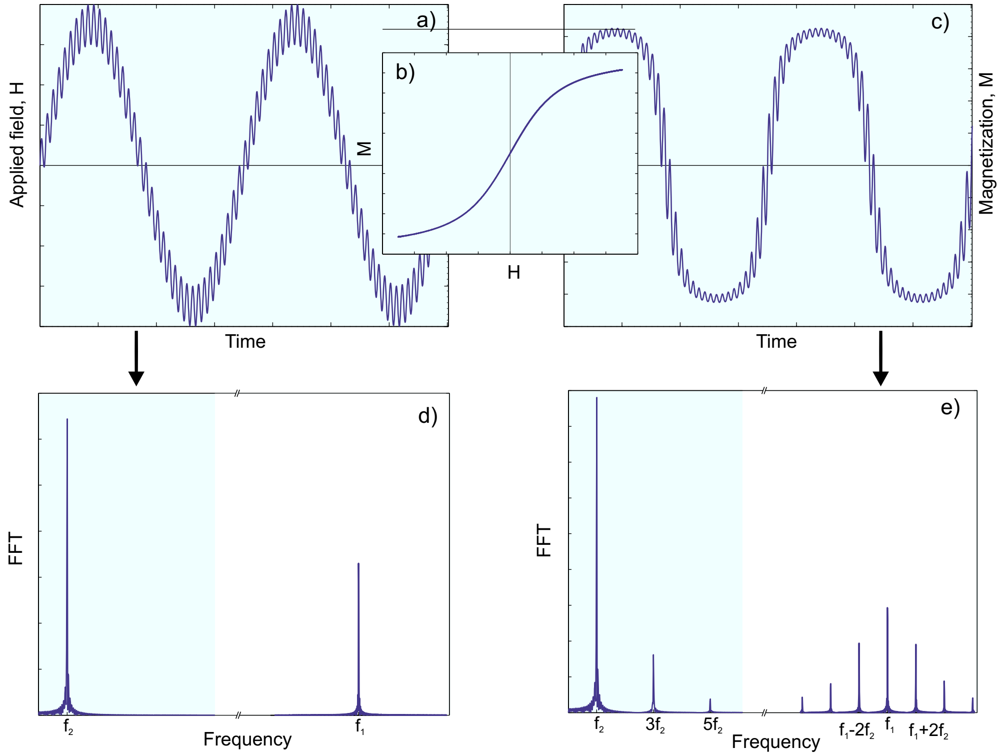

FIG. 1. (Color online) (a) The applied field consists of two frequency components f_1 and f_2, as shown in (a) and (d). Due to the nonlinear magnetization curve of the magnetic sample, (b), the resulting time dependent magnetization (c) shows the frequency mixing components in the frequency spectrum (e).

III. EXPERIMENTAL SETUP

A. Sensor system

To measure the thickness of magnetic layers, a sensor system has been designed according to Fig. 2. Several aspects had to be considered for the development of the system: the amplitude of the low frequency component of the magnetic driving field must be high enough to bring the magnetic material into saturation. Hence, a magnetic core with high permeability ($\mu_r \approx 5500$) was used. The magnetic properties of the core should not influence the signal, i.e., the magnetization curve of the core material has to be linear in the considered range of the magnetic field. A small air gap of about 4 mm in the core is used to generate a homogeneous magnetic field (up to 100 mT) in the region where the magnetic sample is localized. The sensing coil L_1 that is located in center of the air gap and perpendicular to the low frequency component of the field is in close proximity to the sample. Thus, an almost closed magnetic circuit through the sample is realized leading to a high signal-to-noise ratio (SNR) and the influence of the magnetic material on the sensing coil's properties is maximized. Furthermore, the diameter of the measurement coil ($\approx$2 cm) allows for locally restricted measurements within the magnetic sample which is defined by the region where the flux of the sensing coil is guided through the magnetic material. The low frequency

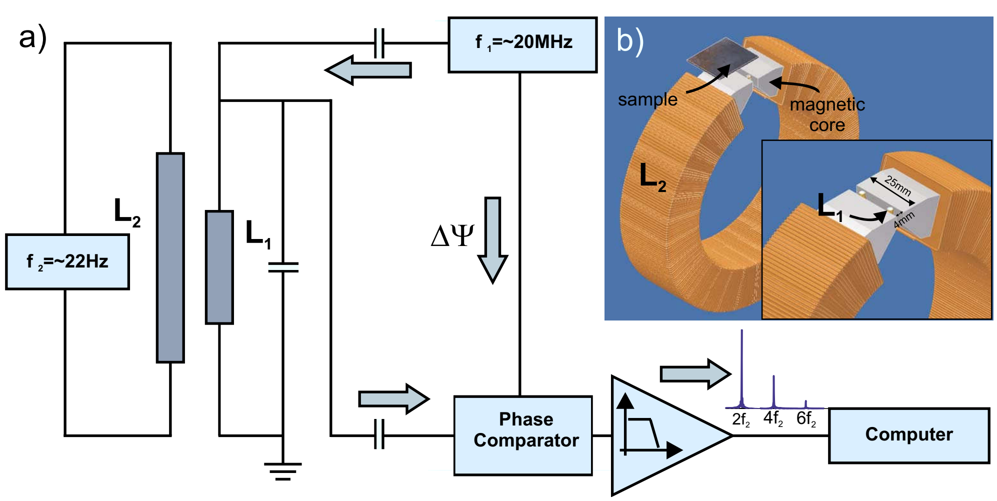

FIG. 2. (Color online) (a) Sensor electronic setup with its main components. The arrows indicates the flow direction of the signals in the circuit. (b) Technical sketch of the coil arrangement.

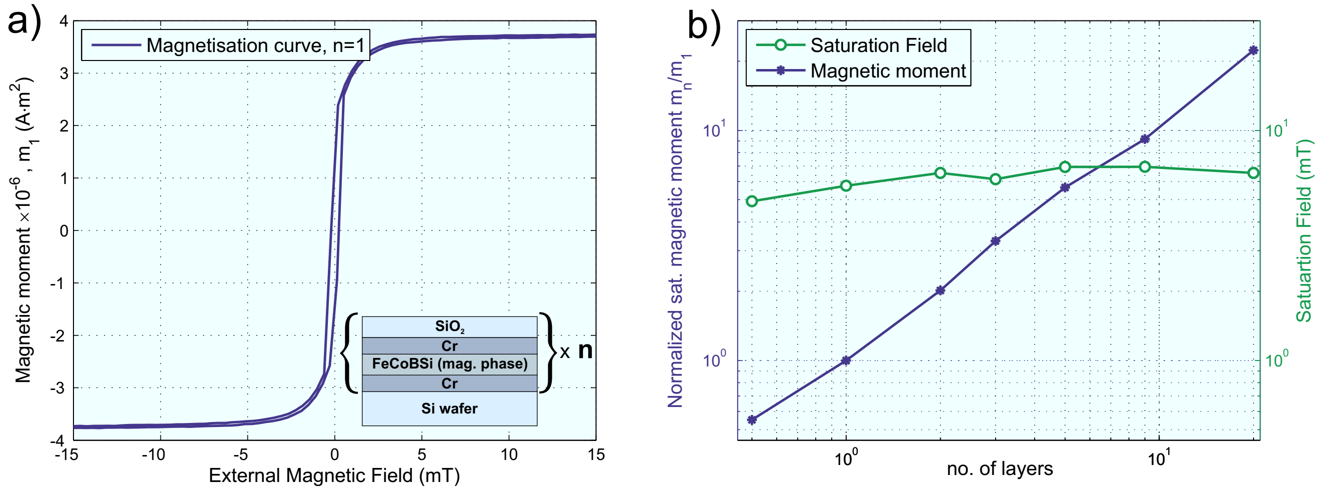

FIG. 3. (Color online) Magnetic properties of multilayer samples used to investigate the sensor performance. As magnetic phase $Fe_{67}Co_{18}B_{14}Si_1$ has been used. (a) The magnetization curve measured using a vibrating sample magnetometer for one layer sample. (b) The dependence of the saturation magnetic moment on the number of magnetic layers is linear, whereas the saturation field remains unchanged.

component of the field (f_2=22 Hz) is realized separately using a signal generator and a low-noise audio amplifier. The high frequency component of the magnetic field (f_1=20.45 MHz) is provided directly by a 2.5 GHz signal generator (IFR 2025).

The frequency mixing signal is measured using a resonant circuit consisting of the sensing coil and a capacitor. The sensing coil L_1 is coupled to the signal generator using a tunable capacitor, as shown in Fig. 2(a). The mixing signal from the magnetic sample is demodulated from the high frequency component using a commercial phase detector (Minicircuits, SYPD-1+). By adjusting the coupling capacities, the phase between the reference (f_1) and the mixing signal ($a\cdot f_1+b\cdot f_2$) without magnetic sample is tuned to $\Delta\Psi=90°$. By this approach, the region of highest sensitivity of the phase comparator is used for the measurements. Furthermore, the signal is low-pass filtered and amplified using an FEMTO system (DLPVA-100-F-S). Further data processing includes A/D conversion (NI DAQPad 6015, 200 kS/s, 16-Bit) followed by spectral analysis calculated using LabView software.

B. Magnetic samples

In order to investigate the sensor performance, samples with defined magnetic properties and thicknesses were fabricated. Samples of different thicknesses have been realized by deposition of metglas (target composition: $Fe_{67}Co_{18}B_{14}Si_1$) on silicon wafers using magnetron sputtering (Von Ardenne, CS730). Since the magnetic properties (hysteresis, permeability) of ferromagnetic films depend generally on the film thickness, multilayers were fabricated [see Fig. 3(a)]. For the present investigation, up to twenty layers have been measured (n=1,2,3,5,9,20). Additionally, a sample was used for the investigation, which was a single layer with half the thickness of the magnetic layer, thus denoted with n=1/2. Only the magnetic phase has been considered for thickness calculation, i.e., the bond coating, insulation layer, and the substrate do not contribute to the sensor signal.

The magnetization curve of a sample with the area of 25 mm^2 and n=1 is shown in Fig. 3(a). The observed magnetic saturation field of about 7 mT is far below the limit of the low frequency magnetic saturation field of the sensor system (100 mT). The dependency of the saturation magnetic moment on thickness is shown in Fig. 3(b). A good linear agreement between thickness, number of layers, and magnetic moment can be observed. A constant saturation field, hysteresis, and permeability have been measured for all samples.

IV. PERFORMANCE AND APPLICATIONS

The performance of the designed sensor is investigated using 13×26 mm^2 multilayer samples with defined thicknesses as described in the previous section. The spectral analysis of the recorded voltage signal is obtained using fast Fourier transformation (FFT).

A FFT spectrum from a magnetic sample consisting of nine layers is shown in Fig. 4(a). It is acquired by averaging over five individual spectra. The background noise signal is subtracted from the spectra in the postprocessing stage. Due to the symmetry and nonlinearity of the magnetization curve, the double value of the driving frequency ($2\cdot f_2$) and its harmonics appear in the spectra. To obtain information about the sample properties, the first four harmonics in the spectrum ($2\cdot i\cdot f_2$, i=1..4), are analyzed. Their amplitudes depend on the properties of the sample, e.g., the amount of the ferromagnetic material. The technique has been limited to the first four harmonics in order to ensure the highest SNR.

In all noncontact magnetic sensor applications, a variation of the air gap between the sensing coil and the magnetic material can lead to an unwanted cross-sensitivity.[15] For this reason, the dependency of the described measurement principle on distance variations was investigated in detail. The amplitude of the measured harmonics in the spectra as function of distance between sensing coil and sample is shown in Fig. 4(b). An exponential decay of the signal amplitude with distance was found. Therefore, by considering only one of

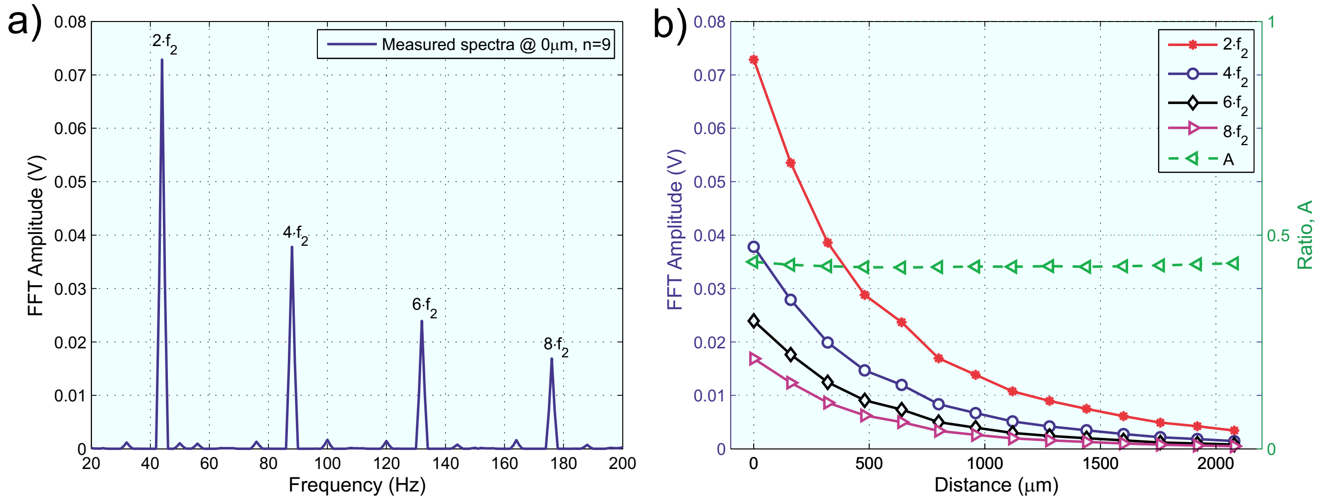

FIG. 4. (Color online) (a) Acquired spectra in contact mode (at 0 μm distance) for a sample with $n=9$ layers. (b) Dependence of the first four harmonics in the FFT spectra on distance. The ratio A (calculated from Eq. (1)) remains unchanged with distance.

these harmonics to measure the amount of magnetic material in the sample, errors can appear due to distance variations between different measurements. For this reason, a ratio of type

$$A = \frac{A_1}{(\Sigma_{i=1}^{4} A_i)^{0.95}}, \quad A_i = \mathrm{FFT}(i \cdot 2 \cdot f_2) \tag{1}$$

is computed. This ratio shows a constant value for a defined sample ($n=9$) while the distance was varied between 0 and 2 mm. Thus, this empirical ratio was considered for all further measurements.

Figure 5 shows the distance independent ratio A as a function of the number of layers. The amount of magnetic material of the coating is directly proportional to the number of multilayers since all the samples have the same lateral dimensions. The results show a logarithmic dependency between the ratio A and the amount of magnetic material of the sample which corresponds to a film thickness range between 55 nm ($n=1/2$) and 2.2 μm ($n=20$). The measured SNR of the 55 nm sample was ≈50 dB. Here, the SNR has been computed as

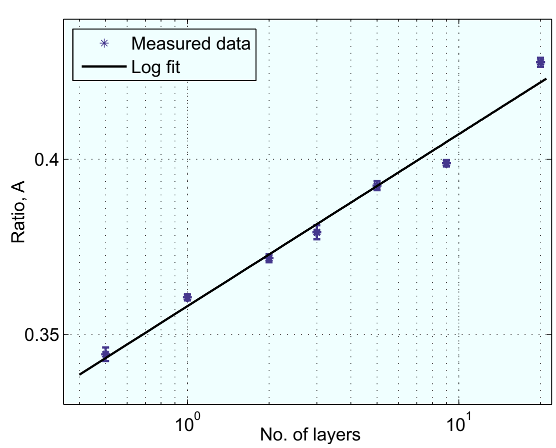

FIG. 5. (Color online) Dependence of the A amplitude on the number of layers of magnetic material (in semilog scales). The error bars denote the standard deviation of repeated independent measurements. The solid line is a logarithmic fit of the data.

$$\mathrm{SNR} = 20 \cdot \log_{10}(\mathrm{FFT}_{+\mathrm{sample}}/\mathrm{FFT}_{-\mathrm{sample}}), \tag{2}$$

where $\mathrm{FFT}_{+\mathrm{sample}}$ and $\mathrm{FFT}_{-\mathrm{sample}}$ are the spectra amplitudes with and without sample, respectively. A logarithmic fit of the measured SNR data indicates that a minimum thickness of approximately 20 nm is measurable, i.e., SNR ≈10 dB.

V. SUMMARY AND CONCLUSIONS

The frequency mixing technique is well established for a long time in sensor applications. Nowadays, it expands to biological and medical applications to detect and track magnetic particles in medical applications. In this paper, the method was employed to measure the magnitude of magnetic moments of thin films and implicitly the thickness of these coatings.

A sensor system based on this method has been designed. The performance of the sensor was tested using magnetic samples with defined properties and thicknesses which were fabricated by deposition of $Fe_{67}Co_{18}B_{14}Si_1$ on silicon wafers using magnetron sputtering. The dependency of the measured signal on the distance between the sensing coil and the sample was investigated. Results have shown an exponential decay of the signal amplitude with the distance. A ratio between the FFT harmonics amplitude has been introduced to allow distance-independent measurements. This way, constant values of the signal at distance variations up to 2 mm were measured. The sensor has been further tested to measure the magnetic properties, i.e., magnetic moment, of samples with different thicknesses. A logarithmic dependence between the signal amplitude and the thickness has been found and demonstrated for thicknesses up to 2.2 μm. Furthermore, due to the high SNR of the method the extrapolated sensitivity corresponds approximately to a magnetic layer thickness of 20 nm.

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