

Non-contact temperature determination of embedded magnetic phases of hard coatings by exploitation of the magnetic hysteresis

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ABSTRACT

A non-contact temperature sensor is described which is based on the temperature dependency of the hysteresis of a magnetic phase embedded in a non-magnetic hard coating. For the measurement principle the technique of frequency mixing is used, with extension toward phase sensitivity as indicator for the temperature. The sensor's capabilities such as selectivity and sensitivity are investigated on TiN–FeCo magnetic multilayers. The sensor has been proven for temperatures up to 500 °C and shows capabilities for higher temperatures.

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1. Introduction

Measurement of physical parameters based on changes of magnetic properties of a sensor phase has already been applied in areas where established methods cannot be used, e.g. extreme temperature applications or UHV [1]. Characteristic examples are represented by pressure and strain sensors, where magnetostrictive materials are used as sensor phase [2]. More recently, superparamagnetic beads as specific labels for proteins or bacteria are used to measure their concentration [3].

To date, for temperature investigation typically the magnetic properties of a sensor phase, i.e. magnetostriction, permeability, or the Curie temperature have been measured as direct indicators [4,5]. More recently, the magnetization curve of superparamagnetic beads and its temperature dependency has been described analytically, enabling their use for temperature measurement in medical applications [6]. In this paper, a new technique based on frequency mixing is proposed. The temperature is indirectly determined by related changes of the magnetic hysteresis of a sensor phase. The performance of the method is tested for temperatures up to 500 °C in the case of TiN-based hard coatings,

where the in-situ non-contact measurement of the temperature of the coating would be a valuable quantity for process optimization.

1.1. Frequency mixing

Frequency mixing has been established as a sophisticated method for the detection of tiny signals from dominating backgrounds [7]. It can be applied if the system to be measured shows a nonlinear response upon excitation. For this an external stimulus is used, which is a superposition of two frequencies f_1, f_2 with $f_1 > f_2$. The high frequency f_1 probes the system's response, while the low frequency f_2 is used to drive the system periodically into its nonlinearity. As a result, the response signal is modulated, generating a defined frequency f_d that can be used for signal detection. In this method, magnetic material with nonlinear magnetization $M(H)$ is exposed to a magnetic field $H(t)$ consisting of two components $H_{1,2}(t) = \hat{H}_{1,2} \sin(2\pi f_{1,2}t)$. An example for such nonlinear M is shown in Fig. 1(a). For $f_1 \gg f_2$ and $\hat{H}_1 \ll \hat{H}_2$, the resulting magnetization in the material is denoted by Fig. 1(b). Here, the field H_2 is chosen high enough to bring the material into saturation ($\hat{H}_2 > H_k$). Under these conditions, the contribution of H_1 to the magnetization M becomes almost zero at maximum value of H_2 . At $H_2 = 0$, the H_1 contribution, as well as the differential susceptibility $\chi = dM/dH$, are maximized.

Due to the symmetry of the magnetization curve, the Fourier spectrum of M shows the odd harmonics of f_2 ($f_2, 3f_2, 5f_2, \dots$), and, more important, the sum frequency mixing terms $f_1 \pm 2nf_2$, $n \in \mathbb{N}$

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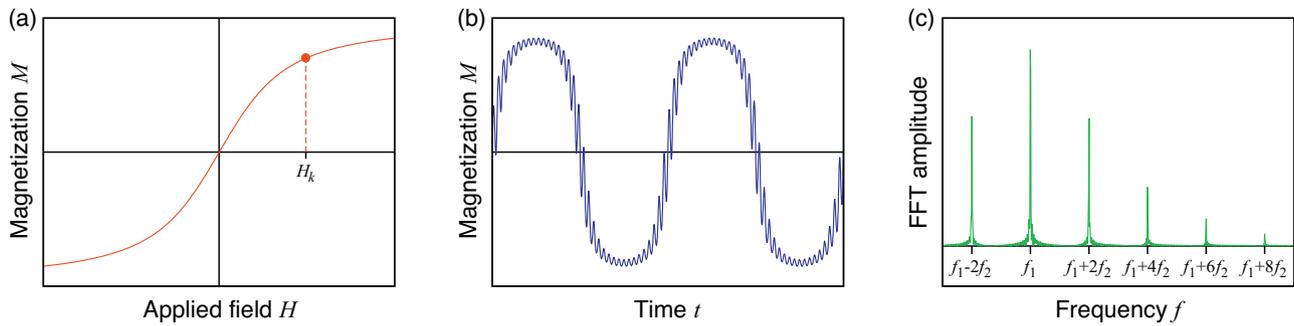


Fig. 1. Frequency mixing principle: (a) magnetization curve $M(H)$ of a material, where H_k denotes a minimum required field to achieve saturation. (b) Magnetization response of the material, if two magnetic fields with frequencies f_1, f_2 are applied. (c) Detail from the resulting FFT spectrum $\tilde{M}(f)$ with the even harmonics at the upper sideband of f_1 .

as upper and lower side bands at the center frequency f_1 . The appearance of these components is highly specific to the nonlinear magnetization curve of the material. Fig. 1(c) shows the upper sideband of f_1 from the Fourier spectrum (FFT) of M , corresponding to the time-dependent magnetization depicted in Fig. 1(b). A detailed description of this principle has been published in previous works [8,9].

The major advantage of this sensing method is its selectivity. The frequency mixing terms are generated only if ferro- or superparamagnetic materials are present. Dia- or paramagnetic material will not produce any frequency mixing signals due to their linear relationship between magnetization and applied magnetic field. As a result, a good signal to noise ratio can be achieved, even if small amounts of ferro- or superparamagnetic material are embedded in a dia- or paramagnetic environment. A linear relation of the magnetic moment and the response signal at $f_1 + 2f_2$ has been demonstrated for magnetic immunoassay [9] and thin film [10] applications.

In recent works, the sum frequency mixing terms have been considered to obtain additional information about the magnetic properties of investigated materials [7]. Teliban et al. [10] have used information from higher mixing terms such as $f_1 + 2nf_2$ with $n > 1$ to eliminate signal fluctuations introduced by variations in distance between the magnetic sensor phase and the detection coils of the non-contact sensor system. Bechtold et al. [11] have demonstrated a strain sensor based on frequency mixing, exploiting the inverse Villari effect of a magnetostrictive material. Here, the modulation field H_2 has been continuously changed to detect characteristic local minima in the even harmonics of the modulation frequency.

All these sensors and their related modeling do not take into account any hysteresis of the magnetic sensor phase. This approach is well-suited since either soft magnetic ferromagnetic materials like Metglas or superparamagnetic materials are used. If this measurement principle can be extended to include the hysteresis, then additional information on the magnetic properties can be gained by this technique, e.g. temperature. Here the method is modified to investigate the phasing of the material's signal response and its implication toward hysteresis and temperature.

1.2. Model for hysteresis detection

The extension of the frequency mixing technique toward hysteresis is achieved by a phase-sensitive detection scheme which is motivated for a model magnetic material. The magnetization curve of materials with ferromagnetic behavior can be approximated by a simplified magnetization curve as shown in solid red in Fig. 2(a). Both regions of magnetic saturation ($|H| > H_k$) are adopted with a slope of zero, i.e. with susceptibility $\chi = 0$. The center region ($|H| \leq H_k$) has a non-zero slope or in terms of susceptibility $\chi = M_s/H_k$. When such a magnetic materials is subjected to an

oscillating external field $H(t)$ with amplitude $\hat{H} > H_k$ as shown in Fig. 2(b), it is periodically saturated. The results are time-dependent responses in magnetization $M(t)$ and susceptibility $\chi(t)$, depicted in Fig. 2(c and d), respectively.

For this model, magnetic hysteresis can be introduced into the magnetization curve by creating two branches for increasing and decreasing field strength with offsets of $\pm H_c$. Fig. 2(a) shows such a magnetization curve as dashed blue lines. In this case the oscillating magnetic field's amplitude \hat{H} needs to exceed $H_k + H_c$ in order to achieve saturation (Fig. 2(b)). The resulting magnetization and susceptibility (Fig. 2(c and d), respectively) are then time-delayed with respect to the response of the magnetization curve without hysteresis. The time delay $\Delta t = t_2 - t_1$ (Fig. 2(b–e), dotted lines) can be detected as a phase shift $\Delta\varphi$ between the response signal and a reference signal. While it is useful to chose the oscillating magnetic field as phase reference, it must be considered that, when monitoring the susceptibility, the fundamental frequency is twice that of the oscillating magnetic field, as shown in Fig. 2(e). That is, a given time delay Δt will correspond to a phase shift $\Delta\varphi_M$ if the magnetization response is used for monitoring and to a phase shift $\Delta\varphi_\chi$ if the susceptibility response is used. Here, for all phase shifts $\Delta\varphi$, the susceptibility is used as reference.

By measuring the phase shift, it is possible to obtain information about the hysteresis properties, i.e. the coercive field H_c . Further changes in the hysteresis, e.g. the slope of magnetization curve, could be also detected similar to approaches used for magnetostrictive sensor phases [11].

2. Experimental setup

2.1. Sample preparation

As test samples, hard coatings with magnetic sensor phase in the form of TiN/FeCo multilayers were fabricated by magnetron sputtering. As substrates, pieces of Si/SiO₂ wafers with lateral dimensions of 5 mm × 30 mm were used. TiN was chosen as a well-known wear resistant hard coating [12], FeCo was used as the magnetic phase due to its soft magnetic and magnetostrictive properties ($\lambda_s = 70$ ppm) and its high Curie temperature [13]. A total coating thickness of 1 μ m was achieved after depositing 178 TiN/FeCo bilayers and one TiN topping layer with single layer thicknesses for TiN and FeCo of 4.2 nm and 1.4 nm, respectively.

Further details of the fabrication and the material's properties were described by Klever et al. [14] The sample's magnetization curves were measured using a Lakeshore Model 7300 vibrating sample magnetometer (VSM) with an additional high temperature oven (Lakeshore 74034) from 30 °C up to 500 °C. Due to sample space limitations in the VSM, the fabricated samples were split into two pieces of 5 mm × 5 mm and 5 mm × 25 mm for usage in the VSM and in the frequency mixing sensor system, respectively.

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