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Sensors and Actuators A 295 (2019) 551–559

Contents lists available at ScienceDirect

Sensors and Actuators A: Physical

journal homepage: www.elsevier.com/locate/sna

Torsional oscillation monitoring by means of a magnetoelastic resonator: modeling and experimental functionalization to measure viscosity of liquids

Luca Lanotte[a,*], Giovanni Ausanio[b,1], Vincenzo Iannotti[b,1], Giovanna Tomaiuolo[c], Luciano Lanotte[d]

[a] INRA, Science et Technologie du Lait et de l'Oeuf 65, rue de Saint Brieuc, 35042 Rennes Cedex, France
[b] CNR-SPIN and Department of Physics "E. Pancini", University of Naples Federico II, Piazzale V. Tecchio 80, I-80125 Naples, Italy
[c] Dipartimento di Ingegneria Chimica, dei Materiali e della Produzione industriale, University of Naples Federico II, Piazzale V. Tecchio 80, I-80125 Naples, Italy
[d] CNR-IPCB and Department of Physics "E. Pancini", University of Naples Federico II, Piazzale V. Tecchio 80, I-80125 Naples, Italy

ARTICLE INFO

Article history:
Received 26 November 2018
Received in revised form 20 May 2019
Accepted 14 June 2019
Available online 21 June 2019

Keywords:
Magnetoelastic resonator
Damped oscillation
Viscosity measurement
Low shear rates

ABSTRACT

A new application of a high sensitivity magnetoelastic resonator able to measure period and damping constant of low frequency torsional oscillation is described and validated by experimental tests. The sensitive parameter is the amplitude of resonant magnetoelastic waves in the soft ferromagnetic core ($Fe_{62.5}Co_6Ni_{7.5}Zr_6Cu_1Nb_2B_{15}$ amorphous ribbon). The theoretical model of the device has been developed, correlating torsional oscillations to the friction force applied by the fluid in which they occur. Thus, an accurate indirect evaluation of fluid viscosity has been demonstrated. The main prerogative of the proposed sensor is to work without contact with the oscillating mechanism. As experimental validation, viscosity of UHT milk was measured versus different fat content. The experimental comparison with a standard rheometer demonstrates the new device competitiveness in the measure of low viscosity fluids at low share rate. Moreover, the detected behaviors at increasing temperature are in agreement with previous literature. In perspective, the new magnetoelastic resonators application can be very ductile and effective in on-line monitoring of viscosity change with time to control composition, degradation or contamination of liquids.

1. Introduction

The property of magnetoelastic amorphous ribbons and wires to change the frequency of electric and/or mechanical resonance as a function of several external parameters (i.e., local magnetic field, tensile stress, temperature, pressure, external loads, damping factors) has been largely employed for measuring static or dynamic strain [1–4], low intensity magnetic field [5], fluid viscosity [6,7], with several applications in industrial engineering [8,9] and bio-medicine [10–12]. In particular, although many alternative techniques are developing for viscosity measurements [13–15], the interest for more reliable and sensitive devices based on magnetoelastic resonance of metallic glass cores is currently alive and fueled by continuous improvements in material treatment [16], device technology [16,17] and measurement methodology itself [18].

Within this framework of research, this study proposes an accurate methodology for torsional oscillation monitoring by means of a magnetoelastic resonator. The sensor working principles and its practical constitution display advantages depending on the use of magnetoelastic wave amplitude at fixed exciting frequency, A_o, as sensitive parameter. The proposed demonstrator consists of a torsional pendulum, ad-hoc conceived and equipped. The measure of the time constant of the damped pendulum oscillation relies on the detection of the oscillation-induced modulation of A_o that reproduces their time behavior. The measure is accurate, reliable and easier especially at low frequency due to the high intensity of the induced modulation. On the other side, we demonstrate that the pendulum oscillation damping depends on the friction forces induced by the viscosity of the fluid in which the pendulum oscillates: in other words, we show that from the measure of A_o damping constant it is possible an indirect measurement of the fluid viscos-

* Corresponding author.
E-mail address: lanotte@fisica.unina.it (L. Lanotte).
[1] These authors contributed equally to this work.

https://doi.org/10.1016/j.sna.2019.06.030
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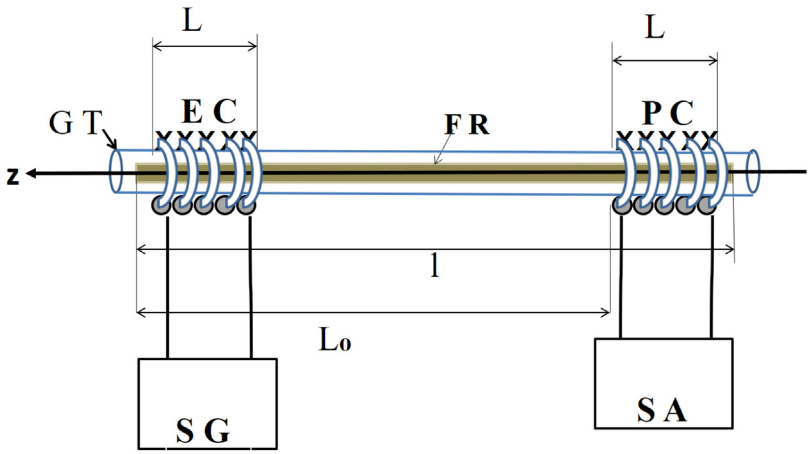

Fig. 1. Scheme of the magnetoelastic resonator. Glass Tube (G T), Ferromagnetic Ribbon (FR), Exciting Coil (EC), Pick-up Coil (PC), Hp Signal Generator (SG), Signal Analyzer (SA).

ity. This represents a significant difference from previous works on coagulation time or viscosity measurement, as the latter generally use the propagation time [19] or, more frequently, the change of resonance frequency and not amplitude of a magnetoelastic wave [6,7,16–18].

The designed final device is able to measure viscosity variations depending on different parameters, such as temperature. Furthermore, the torsional mechanical equipment can be easily inserted into conduits or reservoirs, keeping the sensor core completely outside and without determining contamination. Therefore, the technique appears very attractive in particular for monitoring on-line evolutions with temperature and time degradations due to chemical process.

In the first instance, the components of the experimental apparatus, the preliminary tests on magnetoelastic resonator and torsional pendulum, supplemented by measurements and theory, are detailed. Subsequently, milk viscosity measurements are reported in some practical cases, proving the performance of the new device and its operating principles.

2. Apparatuses and methods

2.1. Detection of rotation by means of resonant magnetoelastic waves

The experimental method is based on the production and characterization of stationary magneto-elastic waves in an amorphous ribbon. The used sensitive parameter is the amplitude (A_o) of the alternate electromotive force induced in a micro-coil detecting the magnetic flux produced by longitudinal stationary magnetoelastic waves.

The scheme of the magnetoelastic micro-resonator (MR), used for this investigation, is shown in Fig. 1. In the exciting coil (EC) an alternate current is controlled in amplitude and frequency v by a signal generator (S G). The current amplitude has been tuned to give a maximum magnetic field of 10 A/m in air (H_{zo}) at the middle point on the coil axis. In this condition, and for a frequency of 1 KHz, the mutual induction between the exciting coil and the pick-up coil (PC) produces an induced electromotive force with amplitude 0.1 mV (10^{-4} V accuracy). An amorphous ferromagnetic ribbon (FR) is placed into the two coils as shown in Fig. 1. The ribbon is protected by a glass tube (GT) and suspended along the coil axis by means of a nylon capillary wire. The electromotive force induced in PC in presence of FR is detected by a signal analyzer (SA). The amplitude A_o is a function of the ribbon core and coils geometry, the external static magnetic field applied on the sensor, as well as the elastic and magnetic properties of the ribbon material. In fact, the strain is linked to local magnetization changes by the magneto-elasticity constitutive equations. The A_o value, even in absence of any magnetizing static field, is strongly dependent on the exciting signal frequency and reaches a maximum at the frequency of the fundamental spontaneous vibration, when stationary acoustic longitudinal waves are established. Some decades ago, the alloy $Fe_{62.5}Co_6Ni_{7.5}Zr_6Cu_1Nb_2B_{15}$ was conceived, produced in the shape of thin ribbon and characterized, as the best soft magnetic material, sensitive to very low magnetic field and therefore optimal as resonator core for the detection of low magnetic field and its changes [20–21]. A proper heat treatment, able both to maximize resonant magnetoelastic waves amplitude, at zero magnetizing field, and to achieve higher sensor sensitivity, also in comparison to the previous most performant amorphous metal (Metglass 2826), was also performed. The use of pre-treated $Fe_{62.5}Co_6Ni_{7.5}Zr_6Cu_1Nb_2B_{15}$ ferromagnetic ribbon allowed to eliminate static field generator in the experimental set-up, thus maximizing the resonator response at zero basic magnetic polarization. The magneto-elastic model for a device of this kind was accurately studied in previous papers [22–23] and the validity of the following relation was demonstrated under resonance conditions at the frequency of the second harmonic of the spontaneous longitudinal oscillation of the ribbon:

$$A_0 = \left(\frac{NS}{L}\right)\left|\left\{\cos\left[\left(\frac{2\pi}{l}\right)(L_0+L)\right] - \cos\left[\left(\frac{2\pi}{l}\right)L_0\right]\right\}\right|\mu_0 H_{z0} k_H^2 \left(\frac{E_H}{\rho}\right)^{1/2} \mu_{rH} \quad (1)$$

where N is the loop number of the PC coil, S is the ribbon core cross-section, L, L_0 and l are the lengths indicated in Fig. 1, μ_o is the magnetic permeability in vacuum, H_{zo} is the amplitude of the exciting field in the EC coil, K_H is the magneto-mechanical coupling factor of the core material, E_H is its Young's modulus at constant field, ρ is the density of the ribbon material and μ_{rH} represents the differential magnetic permeability at constant field.

In the case of the present investigation, the sensor ribbon length was 25 mm and to generate the exciting alternate field was used a Hp signal generator (SG), with an accuracy of 10^{-5} V in amplitude of the exciting signal and of 10^{-2} Hz in its frequency pureness.

The described magnetoelastic resonator (MR) is extremely sensitive to the component of the local magnetic field intensity along the ribbon axis **z**, H_{extz}, since it produces variation in the differential magnetic permeability. This effect is well characterized by the experiment described in Fig. 2. A little NdFeB magnet, with a

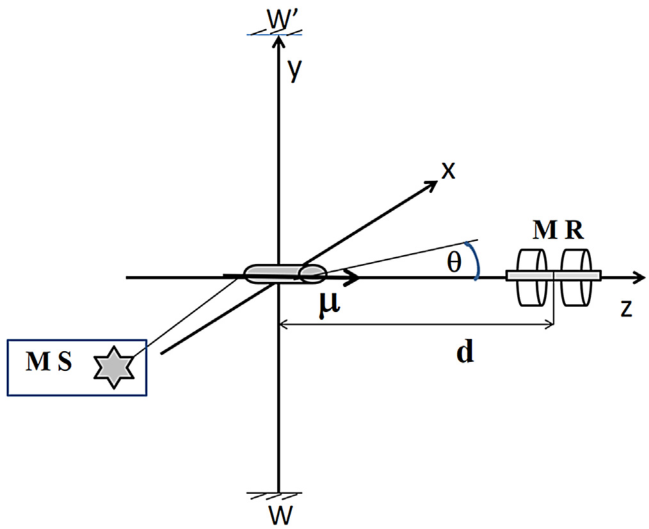

Fig. 2. Experimental arrangement to measure the effect of differential permeability change -induced by variation of the longitudinal component of magnetizing field-on A_o value. Magnetoelastic wave amplitude can be measured as function of both the distance d between the center of the magnetic dipole and the center of the MR sensor, and of the inclination θ of μ with respect to the z axis.

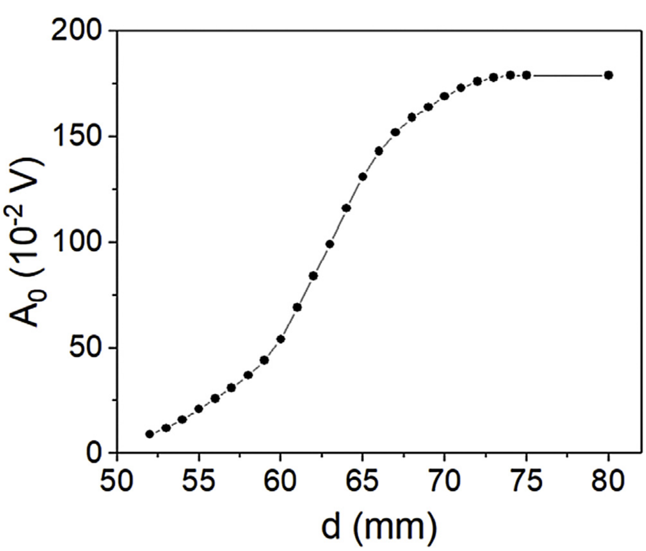

Fig. 3. Amplitude of the magnetoelastic waves detected by the sensor as a function of its distance d from the magnetic dipole (Fig. 2). The working frequency of the magnetoelastic resonator was fixed at the second harmonic of the longitudinal spontaneous oscillation of the ribbon core at zero H field: ν° = 278.04 KHz. From repeated measurements it was found that the amplitude measurements are only affected by the fixed instrumental measurement error equal to ± 3 10^{-3} V, as well as the measure of d presents exclusively an instrumental uncertainty of ±0.25 mm.

magnetic moment μm=25 mA m^2 is placed at a distance d from the middle point of the sensitive core. It is suspended by a nylon thread (W-W') and well balanced to remain in the plane z-x also if a silk wire connected to a micrometric screw (MS) produces a little rotation θ around the y vertical axis.

In this condition, since μ_{rH} decreases with external longitudinal magnetic field (H_{extz}) increment, an increase of A_o with d is expected, until a negligible longitudinal magnetization is imposed on the ribbon core and the magnetoelastic wave amplitude remains unaltered at its value for zero magnetization.

The experimental curve in Fig. 3 validates the prediction and provides also the d value at which the A_o behavior has a derivative maximum: $d_o \approx 63$ mm. In other words, this is the distance at

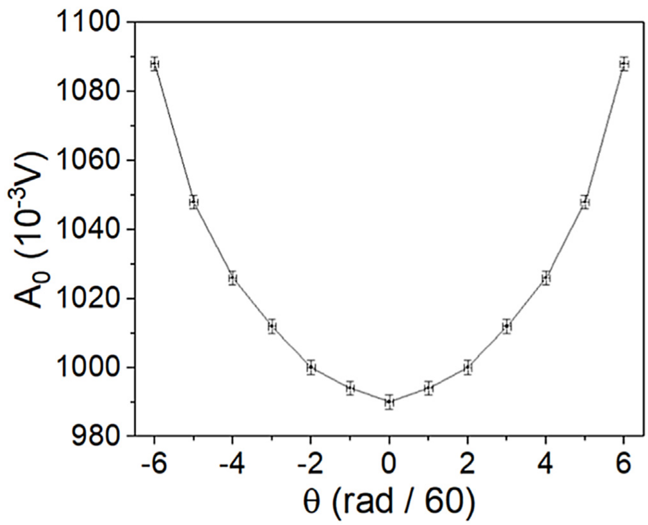

Fig. 4. A_o behaviour versus θ (angle of inclination of the magnetic moment μ with respect to the sensor axis **z** (Fig.2)). On the x axis the angle unit is a minute of radiant (sixtieth of radiant). The error bars have the same amplitude for any experimental point because repeated measurements have always given the same values affected only by the reading error of SA and MS.

which A_o has the maximum sensitivity to any change of longitudinal magnetization.

By inclining the magnetic dipole of an angle θ with respect to the sensor axis (Fig. 2), so that the longitudinal component of the produced magnetic field decreases along z, A_o increases due to the increment of ribbon differential magnetic permeability at lower longitudinal magnetizing field.

The reversible and reproducible curve obtained for A_o vs θ behavior (Fig. 4) constitutes a reliable calibration able to measure θ by A_o evaluation.

It is important to point out that the curve of Fig. 4 is reproduced if the dipole is put in oscillation at low frequency (< 5 Hz). In fact, we are going to demonstrate that this is fundamental to measure the time-depending behavior of slow torsions of any mechanical system, integrated with a magnetic dipole. Moreover, the proposed test for viscosity measurement is just based on the detection of A_o during the oscillation produced on a torsional pendulum immersed in the investigated liquid.

2.2. Experimental device and modeling

Adjustments and additional components were conceived to implement the above described torsion pendulum (Fig. 2) and to develop a theoretical model to predict all the effects which determine its motion, in particular the influence of the viscous forces.

The used prototype apparatus is shown in Fig. 5. A nylon wire is stretched between the points W and W '. Rigidly connected to it, there is a little NdFeB magnet with magnetic moment μ, placed orthogonally to the wire and balanced to rotate in the plane x-z, as in the case of the experiment in Fig. 2. A closed cylindrical bulb (CB) with radius R_1 is connected rigidly to the vertical wire to constitute, together with it and the magnetic dipole, a single torsional pendulum.

The magnetoelastic resonator MR is placed along the z-axis, at a fixed distance d_o = 63 mm, to detects on-line the influence of the axial magnetic field H produced by μ. A solenoid (IF) imposes a field B along x-axis producing a magnetic torque on μ so to induce a fixed initial torsion angle θ○=0.2 rad around y. Once this initial condition has been established, this magnetic field is then kept turned off during the oscillation detection. The CB component is immersed for a depth h in a cylindrical ampoule (CA) of radius R_i, which has

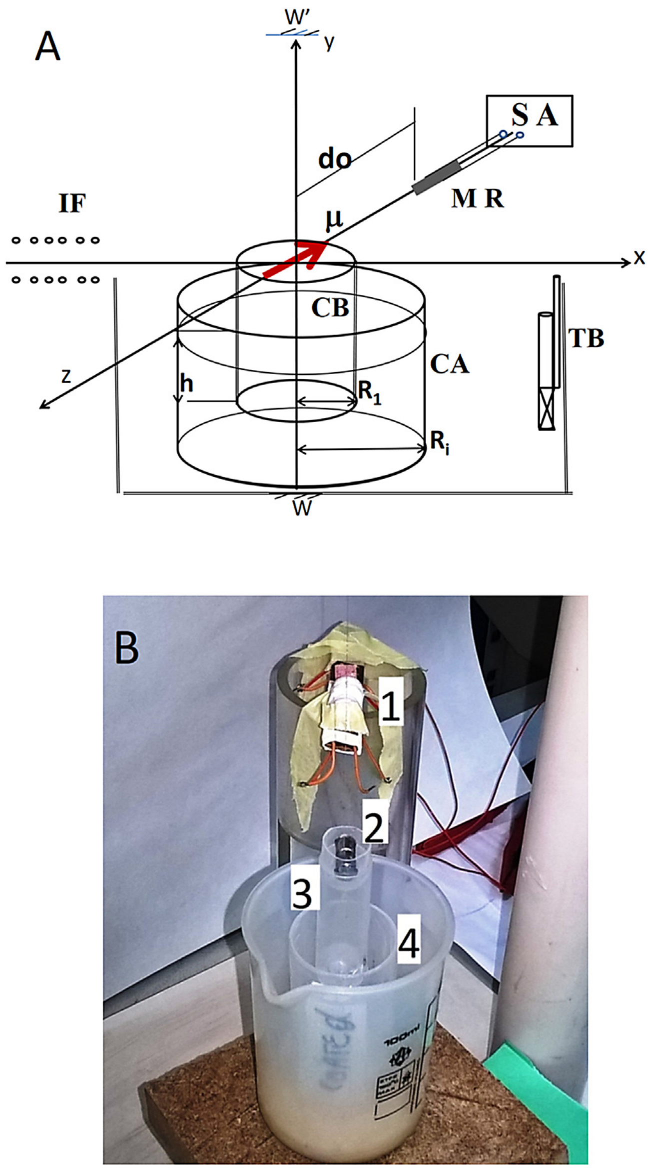

Fig. 5. A. Scheme of the upgraded experimental apparatus: **IF**=solenoid to apply a magnetic field along x axis; $\mathbf{d_o}$=distance between sensor and y vertical axis, coinciding with the oscillation axis of the torsional pendulum; **μ**=magnetic dipole; **MR**=magnetoelastic resonator; **SA**=signal analyzer; **CB**=cylindrical oscillating bulb, integral with the dipole; $\mathbf{R_1}$=radius of internal bulb; **CA**=cylindrical external ampoule; $\mathbf{R_i}$=radius of external ampoule; **TB**=thermal bath equipped with an electric thermostat and a thermometer. **B**. Picture of the first prototype of the upgraded experimental apparatus: magnetoelastic sensor (**1**), magnet dipole (**2**), bulb integral with magnetic dipole (**3**), external fixed ampoule (**4**).

the function to simplifies the modeling of the effect of air viscosity (or any fluid that is poured in the bulb). The device is made so that the radius R_i can be varied, since the fixed base contains different grooves to accommodate ampoules of varous sizes. The experimental setting has been implemented by a thermal bath (TB), equipped with an electric thermostat and a thermometer to perform measurement at controlled temperature (from ambient temperature up to 90 °C).

When the suspended system is left from a fixed initial angle $\theta_\circ$, torsional oscillations occur under the action of the elastic reaction moment, the liquid-induced forces of viscous friction on the CB surface, as well as the friction due to air and clumping mechanism of suspension. For high sensitive measurements the apparatus could be contained in a vacuum chamber and put on an anti-vibration table. The PC coil of the magnetoelastic sensor is connected to a signal analyzer (SA) which detects and visualizes on-line the modulation induced on the resonant wave amplitude, due to the effect discussed in Section 2.1 and shown in Fig. 4.

The equation of motion for the oscillating module, expressed in terms of the temporal variable θ(t) is

$$I_y \frac{d^2\theta(t)}{dt^2} = -K\theta(t) + \eta\left(\frac{dv}{dr}\right)\left(2\pi R_1^2 h + \int_0^{R_1} 2\pi r^2 dr\right) - \gamma\frac{d\theta}{dt} \tag{2}$$

where I_y is the inertial moment of the rotating system, K the elastic torsional constant of the suspension wire, η the viscosity, dv/dr the velocity incremental ratio versus the distance r from the rotation axis y and the term γ dθ(t)/dt includes the effects of friction with air, as well as in the suspension and gluing mechanisms.

In condition $(R_i - R_1) < R_1$, we can consider that v is equal to 0 at distance R_i, on recipient wall, and $(d\theta(t)/dt)\cdot R_1$ on the surface of CB. Thus, assuming a linear velocity decrease, relation (2) can be written as

$$I_y \frac{d^2\theta(t)}{dt^2} = -K\theta(t) - (\eta C + \gamma)\frac{d\theta}{dt} \tag{3}$$

where

$$C = \left\{2\pi\left[\frac{R_1^3}{R_i - R_1}\right]\left(h + \frac{R_1}{3}\right)\right\} \tag{3'}$$

If γ is replaced by η °C, expressing additional frictions through a fictitious viscosity η °C Eq. (3) becomes

$$I_y \frac{d^2\theta(t)}{dt^2} = -K\theta(t) - (\eta + \eta^\circ)C\frac{d\theta}{dt} \tag{4}$$

The general solution of Eq. (4) is

$$\theta(t) = C_1 e^{\alpha t}\cos(\beta t) + C_2 e^{\alpha t}\sin(\beta t)$$

where

$$\alpha = -(\eta + \eta^\circ)\frac{C}{2I_y} \text{ and } \beta = \frac{1}{2I_y}\sqrt{-(\eta + \eta^\circ)^2 C^2 + 4I_y K} \tag{5}$$

By using the following boundary conditions in the point of maximum oscillation velocity (passage through the equilibrium point)

$$t = 0 \Longrightarrow \theta(0) = 0 \text{ and } \frac{d\theta}{dt}(0) = \omega_{maxo}$$

it follows

$$C_1 = 0 \text{ and } C_2 = \frac{\omega_{maxo}}{\beta}$$

Thus the peculiar solution is

$$\theta(t) = \frac{\omega_{maxo}}{\beta} e^{\alpha t}\sin(\beta t) \tag{6}$$

which corresponds to damped harmonic oscillations with a time constant

$$\tau = -\frac{1}{\alpha} = \frac{2I_y}{(\eta + \eta^\circ)C} \tag{7}$$

and period

$$T = \frac{2\pi}{\beta} = \frac{4\pi I_y}{\sqrt{-(\eta + \eta^\circ)^2 C^2 + 4I_y K}} \tag{8}$$

The A_o modulation, due to its proportionality to $\theta^2(t)$ (Fig. 4), decreases both with a time constant and with a period equal to half of that of θ(t): $\tau_* = \tau/2$ and $T^* = T/2$.

Therefore we conclude that, by measuring $A_o(t)$ during torsional oscillation, one expects a periodic and damped modulation of the magnetoelastic waves amplitude, from which it is possible to evaluate the viscosity by measuring the damping constant τ_* or the period T*, using the following equations respectively

$$\eta_\alpha + \eta^\circ = \left[\frac{I_y/C}{\tau^*}\right] \tag{9}$$

$$\eta_\beta + \eta^\circ = \left\{\frac{1}{C}\sqrt{\frac{-4\pi^2 I_y^2}{T^{*2}} + 4I_y K}\right\} \tag{10}$$

Once the fictitious viscosity η° has been estimated, Eqs. (9) and (10) are the constitutive relations of the new proposed device for viscosity measurements. The possibility of changing the constant C by means of R_i variation, i.e. the change of the velocity gradient into the fluid, enables to check if the investigated liquid behaves as a Newtonian one or not. Although both relations appear validly applicable, it is evident that Eq. (9) involves fewer parameters. Moreover, τ_* value is expected higher than T* one, therefore it is evaluable with higher relative precision. Standing these considerations, we have used the Eq. (9) for the viscosity measurement.

3. Experimental calibration and tests

3.1. Calibration using distilled water as reference liquid

The first prototype of the experimental apparatus is represented in Fig. 5B. The values of the geometrical parameters are: $R_i = 1.2{\cdot}10^{-2}$ m; $R_1 = 0.7{\cdot}10^{-2}$ m and $h = 2.0{\cdot}10^{-2}$ m. Therefore, from Eq. (3') it follows that $C = 9.6\ 10^{-6}\ m^3$. The moment of inertia I_y, as well as of fictitious viscosity η° are not easily determinable by direct calculation as it is complicated to take into account of the mass distribution and the friction related to the various elements of the oscillating equipment: magnetic dipole, CB, nylon thread, fixing glue etc. Therefore, in order to obtain the values of η° and I_y the procedure described below was adopted.

At first, the torsional oscillation is produced in air ($\eta_\alpha = 0$ in Eq. (9)). Fig. 6 above reproduces the photo of the signal analyzer screen. The signature of both resonant wave and its damped periodic modulation induced by the magnetic dipole vibration is evident. By subtracting the fixed resonant wave component from the S A acquired data, the corresponding plot of the only modulating component due to the damped oscillation of the magnetic dipole is easily obtained as shown in Fig. 6 below. The constant of time τ^* has been estimated by individuating the modulating signal maxima and the logarithmic decrement of their analytic best fit. The average value, obtained from reiterated measurements, was $\tau^\circ = (21.8 \pm 0.5)$s.

Secondly, CB oscillations are produced in distilled water at room temperature (20 °C). The corresponding damped oscillation signal from the sensor is plotted in Fig. 7. The elaboration by reiterated measurements provides the average value of the damping constant $\tau^w = (1.28 \pm 0.03$s.

When Eq. (9) is applied to the measurements in air and in distilled water, two analytical relationships are obtained:

$$\eta^\circ = \frac{I_y/C}{\tau^\circ};\ \eta^w + \eta^\circ = \frac{I_y/C}{\tau^w} \tag{11}$$

Replacing the measured values of τ° and τ^w, and the known value of viscosity of distilled water at room temperature, $\eta^w = 1$ mPa s, in Eq. (11) it is easy to calculate the remaining parameters necessary to use Eq. (9) for the measurement of the unknown viscosity, so obtaining: $\eta^\circ = (64 \pm 4)\ 10^{-6}$ Pa·s and $I_y = (13.4 \pm 10^{-9}\ Nms^2$.

Such a procedure used to determine the fictitious viscosity and the moment of inertia represents a sort of calibration of any experimental apparatus realized on the basis of the methodology here

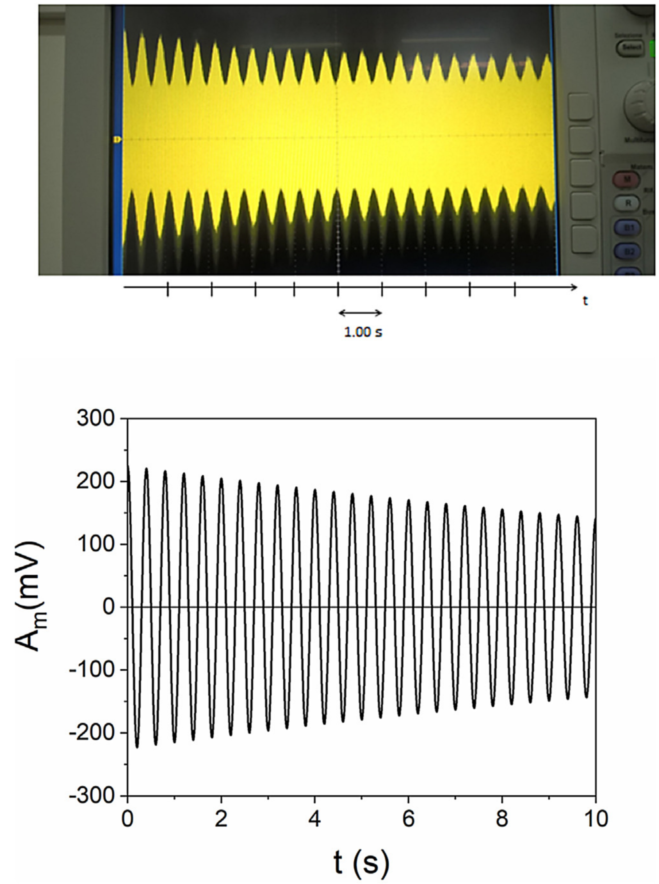

Fig. 6. Modulation of magnetoelastic wave amplitude induced by oscillation of torsional pendulum in air as seen from oscilloscope screen (above) and as plotted (below, amplitude A_m versus time t) after extracting from the acquired data the modulating component induced by the damped oscillations of the pendulum (by simple subtraction of the bearing component, with constant amplitude and at a higher frequency, due to the resonant magnetoelastic wave). From this plot the intrinsic fictitious viscosity η° has been measured by means of the signal maxima exponential, decrement according to the result of the theoretical model contained in Eq. (9).

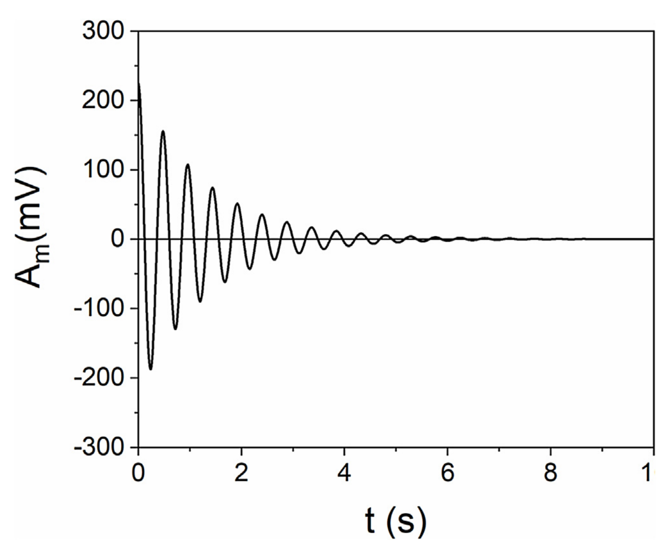

Fig. 7. Amplitude A_m versus time t of the resonant magnetoelastic wave modulation as elaborated by the signal analyzer. The logarithmic decrement can easily derived from the values at the maximum points.

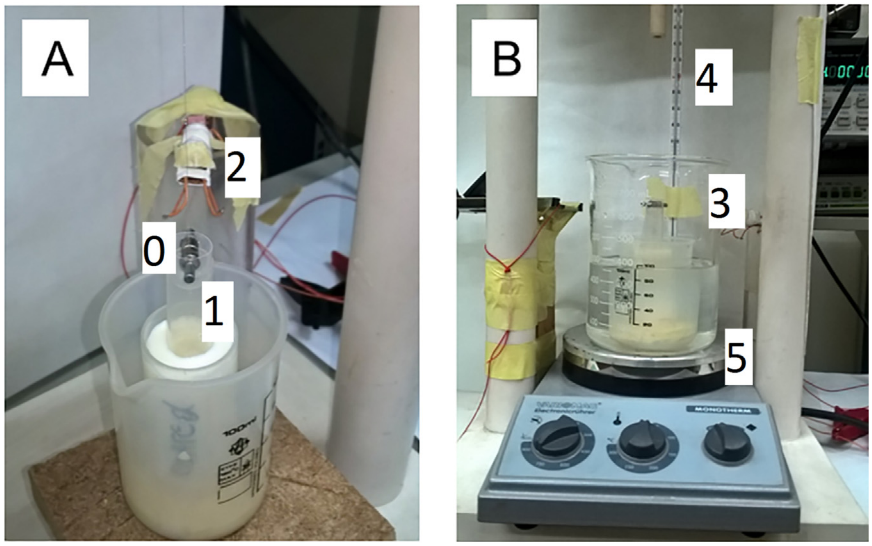

Fig. 8. **A**- Experimental apparatus with magnetic dipole (**0**), oscillating element immersed in milk (**1**) and sensor (**2**). **B**- Apparatus A immersed in a simple thermostatic cell, constituted by a pyrex ampoule (**3**), a thermometer (**4**) and an hotplate (**5**), using water to stabilize thermal exchange.

proposed. It must be repeated if some element of the torsion pendulum has been for any reason modified or changed.

The η° found in this practical case is appropriate because it is negligible compared to the expected viscosity values of the liquids for which the apparatus is designated. It constitutes a corrective term to be considered for the exact determination of viscosity by means of Eq. (9). On the other hand, this empirical calibration incorporates approximations present in the model making them not crucial in viscosity sensing applications.

3.2. Tests on milk

To verify the reliability of the proposed device, we performed tests on commercial milk (Fig. 8A). This liquid food is appropriate for a basic investigation because its properties were fully investigated by previous literature. Moreover, milk undergoes appreciable viscosity change due to transformation processes (e. g. rennet addition), temperature increment or standard processes (skimming, acidification, protein addition, etc.) and dangerous effects induced by adulteration or forbidden additives [24–28].

Therefore, monitoring the viscosity of milk (η^m) is essential to evaluate and protect the genuineness of such an important and nutritious element. For this run of experiments the ampoules in Fig. 8A have been inserted into a thermostat, as shown in the image in Fig. 8B. Starting from 20 °C, temperature was increased by regular fixed steps and the viscosity measurements were performed after the new value stabilization. The fully skimmed, partially skimmed and whole long-life UHT milk has been investigated immediately after opening the container. The aim is to verify the expected decrease of viscosity due to the reduction of the fat content and the increase in temperature.

On the basis of Eq. (9), it is necessary to increase I_y to work at a higher value of τ_*. Since we expect viscosity values in milk between 1 mPa·s and 2 mPa·s, the inertial moment has been preliminarily increased so as to ensure that τ_* assumes corresponding values above one second. This was obtained by inserting small masses inside the cylindrical oscillating component, not altering the friction with the air and other effect that could change η°, and measuring the new value of τ^w. Since we calculated $\tau^{w'}$ = (4.35 ± 0.09)s the new moment of inertia I_y'= (45.7 ± 1.0)·10^{-9} Nms^2 has been obtained.

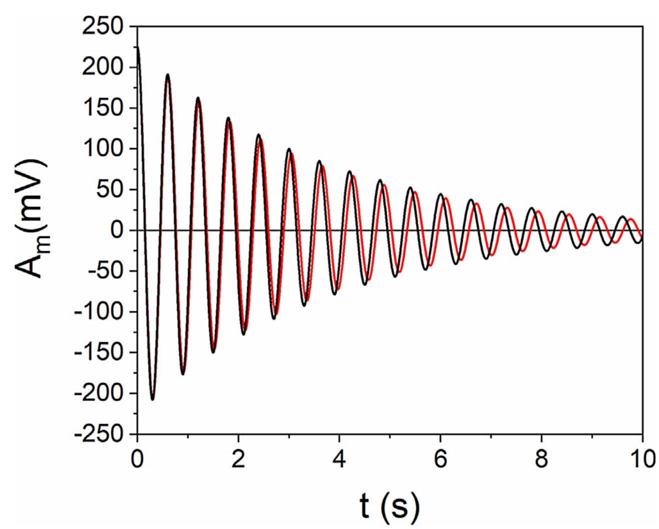

Fig. 9. Attenuation timeline of the amplitude Am of the modulation induced by the pendular damped oscillations on the magnetoelastic wave, as detected by the sensor at 20 °C (black line) and at 50 °C (red line, shifted to the right) in the same boundary conditions and in the same sample of skimmed UHT milk. It appears the decrement of damping of torsional oscillation when temperature increases. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article).

Fig. 9 illustrates the exponential reduction of magnetic wave modulation detected by the magnetoelastic sensor as consequence of the damped oscillation of the pendulum in skimmed UHT milk, at 20 °C (image on the left) and 50 °C (image on the right). The increment of the time constant τ_* due to the increase of temperature, and consequently the decrease of viscosity induced by heating, is evident.

In Table 1, the milk characteristics, and the corresponding value of τ_* and of the viscosity η^m, are reported in detail as a function of temperature in the range from 20 °C to 50 °C.

It is possible to deduce an η^m increase of 21% obtained with fraction of fat, at room temperature, and a decrease in viscosity induced by temperature at any fat percentage. These behaviors are consistent with the results obtained in previous literature on viscosity properties of milk [29–31]. Therefore, the experimental results summarized in Table 1 support the reliability of the new proposed

Table 1
Time constant and viscosity measured by the prototype device as a function of UHT milk fat content and temperature.

Milk type	Fat content	$\tau*(20\,^{\circ}C)$(s)	$\eta^m(20\,^{\circ}C)$ (mPa·s)	$\tau*(30\,^{\circ}C)$ (s)	$\eta^m(30\,^{\circ}C)$ (mPa·s)	$\tau*(40\,^{\circ}C)$ (s)	$\eta^m(40\,^{\circ}C)$ (mPa·s)	$\tau*(50\,^{\circ}C)$ (s)	$\eta^m(50\,^{\circ}C)$ (mPa·s)
Whole UHT milk	3.5 g/100ml	2.86 ± 0.05	1.87 ± 0.05	3.22 ± 0.06	1.6 8 ± 0.04	3.49 ± 0.06	1.57 ± 0.04	3.60 ± 0.06	1.53 ± 0.04
Partially skimmed UHT milk	1.6 g/100ml	3.15 ± 0.04	1.72 ± 0.04	3.37 ± 0.04	1.52 ± 0.03	3.55 ± 0.05	1.55 ± 0.03	3.63 ± 0.05	1.52 ± 0.03
Skimmed UHT milk	0.1 g/100ml	3.53 ± 0.05	1.55 ± 0.03	3.59 ± 0.05	1.53 ± 0.03	3.64 ± 0.05	1.51 ± 0.02	3.68 ± 0.05	1.50 ± 0.03

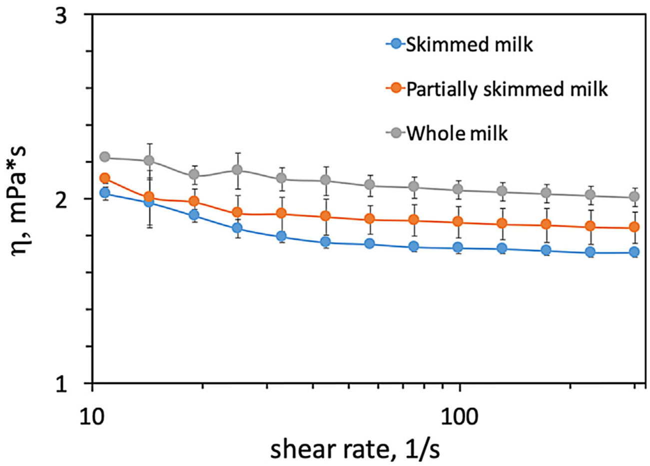

Fig. 10. Viscosity measured by rotational viscosimeter versus shear rate, at 20 °C (log plot for x axis). Average value and related standard deviation are: 2.05 mPa·s ± 0.05 mPa·s (whole milk, upper line), 1.87 mPa·s ± 0.08 mPa·s (partially skimmed milk, middle line) and 1.73 mPa·s ± 0.03 mPa·s (skimmed milk, lower line).

application of magnetoelastic sensors, as well as the effectiveness of the exposed theoretical model of the experiment.

Nonetheless, to have a further direct experimental support, comparative measurements at room temperature by means of a standard instrument were also performed.

A stress-controlled Physica rheometer MCR 301 (Anton-Paar, Graz, Austria) equipped with a titanium double-gap measuring system (DG 26.7, inner cup diameter 24.267 mm, inner bob diameter 24.666 mm, outer bob diameter 26.663 mm, outer cup diameter 27.053 mm, and bob height: 40.000 mm) has been used. The bob rotates inside the cup, which is still and maintained at a constant temperature of 20 °C by a Peltier system. This assembly, presenting a high surface area and requiring small amount of sample (i.e., 1.8 ml), is ideal for low viscous materials, such as milk.

The following salient conclusions are deduced from the data shown in Fig. 10: i) the Newtonian nature (i.e. viscosity is constant with shear rate) of cow milk is confirmed; ii) the decrease of viscosity by decreasing fat content is coherent with the behavior evidenced by magnetoelastic sensor measurements; iii) the values obtained are systematically higher by about 10% compared to those correspondingly measured with the new methodology proposed here (this is acceptable and probably due to some systematic difference in the calibration parameter as C, η° . . .).

In any case, it is particularly important that the proposed viscosity measurement method, despite working at low shear rates (around 1/s), provides values and trends similar to those of the rotational viscometer, which, however, does not provide reliable values below 10/s.

Indeed, the measurement of such low viscosity values, although performable in principle with commercially-available rotational rheometers under specific configurations and operating conditions, is actually a very challenging task. This can be appreciated in Fig. 10 where only after 10/s the instrument starts providing reliable data (viscosity values below the measurement range of the rheometer are not shown for the sake of clarity).

Therefore, the new magnetoelastic resonator application appears very efficient to study the behavior of low viscosity fluids at low shear rate, providing much more reliable viscosity data at low shear rates as compared to classical rheometers. In particular, this ability represents a potential powerful tool for the analysis of non-Newtonian fluids, such as blood [32] as well as other low-viscosity complex fluids (i.e. attractive suspensions [33] and emulsions [34]). In fact, in such fluids the shear thinning behavior is mostly related to aggregation and structuring phenomena that occur at low shear rates. Other fluids for which low shear rate measurements are important are active matter suspensions, as bacteria suspension, where viscosity measurements in the dilute and semi-dilute regime are fundamentals for evidencing a kind of superfluid transition [35]. However, as stated previously, reliable measurements of viscosity at very low shear rates, as the ones achievable by the proposed method (i.e. 1/s) are challenging in classical rheometers.

4. Conclusions

On the basis of previous studies, exploiting the extraordinary softness of the ferromagnetic alloy $Fe_{62.5}Co_6Ni_{7.5}Zr_6Cu_1Nb_2B_{15}$, we succeeded in realizing a resonator in which the magnetoelastic wave amplitude reaches record values in very low magnetizing field, decreasing rapidly when this field is increased. Using this fundamental property, we have developed theory and application of the optimized magnetoelastic resonator. The results below listed can be claimed.

1st - The torsion angle of a pendulum, equipped with a magnetic dipole, has been monitored with high sensitivity by variation of the local longitudinal magnetization component applied to the sensor core.

2nd – It has been demonstrated that the torsion angle, during the oscillation of the device in a fluid, follows an equation of motion whose solution provides a decreasing exponential decrement of the maxima of the oscillation amplitude. Moreover the relative time constant results closely related to the geometry of the mechanical element under torsion, to its moment of inertia and, in particular, to the viscosity of the fluid in which the damped oscillation occurs. In this way, the constitutive equation of the viscosity measuring device was established.

3rd - The procedure of calibration to determine the unknown parameters in the model has been accurately explained and simplified, thus making easy the application correspondent to the various practical cases. In fact, experimental tests have been reported confirming the sensitivity and reliability of the new device compared to the temperature effects already highlighted by previous authors.

4th – Finally, the high sensitivity of the proposed viscositymeter, in particular at low shear rate values (< 10/s), demonstrates its high competitiveness compared to standard rotational rheometers for low viscosity measurements.

The device is very ductile: (i) it is possible to monitor low frequency oscillation performing accurate evaluation of damping constant and period; (ii) it is easy to change the measure range by modifying the moment of inertia of the torsion pendulum; (iii) investigation can be performed as a function of temperature; (iv) for viscosity evaluation the oscillating element can be placed in the fluid to be monitored by keeping the resonator outside, thus avoiding any disturbance or contamination of the liquid due to the sensor; (v) by changing radius of the twisted element CB and of the ampoule containing it, it is possible to change the shear rate and therefore also to detect non-Newtonian effects.

The experiment model developed in the case of an oscillating pendulum incorporates the presence of eventual neglected effects by means of fictitious parameters and appears efficient and relevant for a lot of practical cases. In particular, looking to guidelines for further research, the proposed device offers a potential solution for reliable measurements in low viscosity liquids at low share rate which are generally very complicated using conventional rheometers. As an example, it appears very interesting to address the short-term investigation to the study of blood viscosity as a function of thinners content, at a low flow rate regime, since these conditions are interesting for the prevention of problems such as stroke, palpitations, thrombosis, heart attacks and hypertension [36,37].

Acknowledgements

The authors are grateful to Stefano Marrazzo (mechanical technician at the Department of Physics, University of Naples Federico II) for his collaboration in realizing some components of the experimental apparatuses. This work was supported through laboratory materials and instruments by Department of Physics E. Pancini, and Dipartimento di Ingegneria Chimica, dei Materiali e della Produzione Industriale, University of Naples Federico II.

References

[1] K.-H. Shin, M. Inoue, K.-I. Arai, Strain sensitivity of highly magnetostrictive amorphous films for use in microstrain sensors, J. Appl. Phys. 85 (1999) 5465–5467, http://dx.doi.org/10.1063/1.369977.
[2] G. Ausanio, V. Iannotti, C. Luponio, L. Lanotte, R. Germano, A. D'Agostino, M. Inverno, R. Sorrentino, Potential applications of innovative magnetoelastic resonators for vibration detection, Sens. Actutors A 91 (2001) 123–125, http://dx.doi.org/10.1016/S0924-4247(01)00474-5.
[3] C. Gomez-Polo, J.M. Barandarian, J. Gutierez, New field/position detection method based on a metallic resonant element, Sens. Actuators A: Phys. 106 (2003) 155–158, http://dx.doi.org/10.1016/S0924-4247(03)00155-9.
[4] A. Dalponte, E.S. Bastos, F.P. Missel, Enhanced response from field-annealed magnetoelastic strain sensor, J. Appl. Phys. 120 (2016), http://dx.doi.org/10.1063/1.4960687.
[5] G. Ausanio, V. Iannotti, L. Lanotte, Giant magnetic induction in magneto-elastic resonators and its application for magnetic field sensor, Sens. Actuators A Phys. 153 (2009) 162–165, http://dx.doi.org/10.1016/j.sna.2009.04.034.
[6] P. Cheng, S. Gao, W. Zhang, T. Wang, A. Jian, S. Sang, Resonance modes of free standing magnetoelastic resonator and the application in viscosity measurement, Smart Mater. Struct. 24 (2015), http://dx.doi.org/10.1088/0964-1726/24/4/045029.
[7] S. Atalay, V.S. Kolat, N. Bayri, T. Izgi, Magnetoelastic sensor studies on amorphous magnetic FeSiB wire and the application in viscosity measurement, J. Supercond. Nov. Magn. 29 (2016) 1551–1556, http://dx.doi.org/10.1007/s10948-016-3440-3.
[8] J.S. Choi, J.W. Yoo, S.K. Kauh, Modeling of magnetoelastic resonator using h-parameter analysis, J. Mech. Sci. Technol. 30 (2016) 749–766, http://dx.doi.org/10.1007/s12206-016-0130-9.
[9] S. Sang, S. Gao, X. Guo, P. Cheng, W. Zhang, The detection of Pb^{2+} in solution using bare magnetoelastic resonator, Appl. Phys. Lett. 108 (2016), 054102, http://dx.doi.org/10.1063/1.4941024.
[10] J. Wam, M.L. Johnson, R. Guntupalli, V.A. Petrenko, B.A. Chin, Detection of Bacillus anthracis spores in liquid using phage-based magnetoelastic micro-resonators, Sens. Actuators B Chem. 127 (2007) 559–566, http://dx.doi.org/10.1016/j.snb.2007.05.017.
[11] L.V.R. Beltrami, M. Beltrami, M. Roesch-Ely, S.R. Kunst, F.P. Missel, E.J. Birriel, C. de F Malfatti, Magnetoelastic sensors with hybrid films for bacteria detection in milk, J. Food Eng. 212 (2017) 18–28, http://dx.doi.org/10.1016/j.jfoofeng.2017.05.010.
[12] A. Sagasti, J. Gutierrez, M.S. Sebastian, J.M. Barandarian, Magnetoelastic resonators for highly specific chemical and biological detection: a critical study, IEEE Trans. Magnet. 53 (2016), http://dx.doi.org/10.1109/TMAG.2016.2622359.
[13] K. Waszczuk, T. Piasecki, K. Nitsch, T. Gotszalk, Application of piezoelectric tuning forks in liquid viscosity and density measurements, Sens. Actuators B Chem 160 (2011) 517–523, http://dx.doi.org/10.1016/j.snb.2011.08.020.
[14] Yu Hongbin, Lou Liang, Alex Gu Yuandong, Capacitive micromachined ultrasonic transducer (CMUT) based micro viscosity sensor, Sens. Actuators B Chem. 227 (2016) 346–351, http://dx.doi.org/10.1016/j.snb.2015.12.061.
[15] Sein Oh, Byeongyeon Kim, Jeong K. Lee, Sungyoung Choi, 3D-printed capillary circuits for rapid, low-cost, portable analysis of blood viscosity, Sens. Actuators B Chem. 259 (2018) 106–113, http://dx.doi.org/10.1016/j.snb.2017.12.025.
[16] A. Garcia-Arribas, J. Gutierrez, G.V. Kurlyandskaya, J.M. Barandiaran, A. Svalov, E. Fernandez, A. Lasheras, D. de Cos, I. Bravo-Imaz, Sensor applications of soft magnetic materials based on magneto-impedence, magneto-elastic resonance and magneto-electricity, Sensors 14 (2014) 7602–7624, http://dx.doi.org/10.3390/s140507602.
[17] P. Chen, Q. Jiang, S. Horikawa, S. Li, Magnetoelastic-sensor integrated microfluidic chip for the measurement of blood plasma viscosity, J. Eletrochem. Soc. 164 (2017) B247–B252, http://dx.doi.org/10.1149/2.1441706jes.
[18] S. Karuppuswami, A. Kaur, H. Arangali, P.P. Chahal, A hybrid magetoelastic wireless sensor for detection of food adulteration, IEEE Sens. J. 17 (2017) 1–15, http://dx.doi.org/10.1109/JSEN.2017.2656476.
[19] E. Maliaritsi, L. Zoumpoulakis, J. Simitzis, P. Vassiliou, E. Hristoforou, Coagulation sensors based on magnetostrictive delay lines for biomedical and chemical engineering applications, J. Magn. Magn. Mater. 299 (2006) 41–52, http://dx.doi.org/10.1016/j.jmmm.2005.03.095.
[20] L. Lanotte, G. Ausanio, M. Carbucicchio, V. Iannotti, M.Müller, Coexistence of very soft magnetism and good magnetoelastic coupling in the amorphous alloy $Fe_{62.5}Co_6Ni_{7.5}Zr_6Cu_1Nb_2B_{15}$, J. Magn. Magn. Mater. 215-216 (2000) 276–279, http://dx.doi.org/10.1016/S0304-8853(00)00133-5.
[21] G. Ausanio, V. Iannotti, C. Luponio, L. Lanotte, R. Germano, A. D'Agostino, M. Inverno, R. Sorrentino, Potential application of innovative magnetoelastic resonators for vibration detection, Sens. Actuators A Phys. 91 (2001) 123–125, http://dx.doi.org/10.1016/S0924-4247(01)00474-5.
[22] V. Iannotti, L. Lanotte, Modelling of the magnetoelastic wave amplitude in amorphous ferromagnetic ribbons, Philos. Mag. B 80 (2000) 1903–1920, http://dx.doi.org/10.1080/13642810008216513.
[23] G. Ausanio, V. Iannotti, L. Lanotte, Giant magnetic induction in magneto-elastic resonators and its application for magnetic field sensors, Sens. Actuators A Phys. 153 (2009) 162–165, http://dx.doi.org/10.1016/j.sna.2009.04.034.
[24] A. Bienvenue, R. Jiménez-Flores, H. Singh, Rheological properties of concentrated skim milk: influence of heat treatment and genetic variants on the changes in viscosity during storage, J. Agric. Food Chem. 51 (2003) 6488–6494, http://dx.doi.org/10.1021/jf034050+.
[25] Panagiotis Sfakianakis, Evangelos Topakas, Constantina Tzia, Comparative study on high-intensity ultrasound and pressure milk homogenization: effect on the kinetics of yogurt fermentation process, Food Bioprocess. Technol. 8 (2015), http://dx.doi.org/10.1007/s11947-014-1412-9.
[26] G. Kyazze, V. Starov, Viscosity of milk: influence of cluster formation, Colloid J. 66 (2004) 316–321, http://dx.doi.org/10.1023/B:COLL.0000030842.70947.f0.
[27] R. Stephani, A. Borges de Souza, M.A. Leal de Oliveira, I.T. Perrone, A. Fernandes de Carvalho, Evaluation of synergistic effects of milk protein in rapid viscosity analyzer, J. Diry Sci. 98 (2015) 8333–8347, http://dx.doi.org/10.3168/jds.2015.9300.
[28] S.G. Sutariya, T. Huppertz, H.A. Patel, Influence of milk pre-heating conditions on casein-whey protein interactions and skim milk concentrate viscosity, Int. Dairy J. 69 (2017) 19–22, http://dx.doi.org/10.1016/j.idairyj.2017.01.007.
[29] A.S. Bakshi, D.E. Smith, Effect of fat content and temperature on viscosity in relation to pumping requirements of fluid milk products, Meet. Abstr. Am. Dairy Sci. Assoc. 67 (1984) 1157–1160, http://dx.doi.org/10.3168/jds.S0022-0302(84)81417-4.
[30] L.A. Pereira Alcantara, R. Da Costa Ilhéu Fontan, R.C. Ferreira Bonomo, E. Cardozo de Souza Jr., V. Santos Smpaio, R. Goncalves Pereira, Density and dynamic viscosity of bovine milk affect by temperature and composition, Int. J. Food Eng. 8 (2012) 11, http://dx.doi.org/10.1515/1556-3758.1860.
[31] V. Kumbár1, Š. Nedomová, Viscosity and analytical differences betweem raw milk and UHT milk of Czech cows, Sci. Agric. Bohem. 46 (2015) 78–83, http://dx.doi.org/10.1515/sab-2015-0020.
[32] G. Tomaiuolo, A. Carciati, S. Caserta, S. Guido, Blood linear viscoelasticity by small amplitude oscillatory flow, Rheol. Acta 55 (2016) 485–495, http://dx.doi.org/10.1007/s00397-015-0894-3.
[33] N.J. Wagner, J.F. Brady, Shear thickening in colloidal dispersions, Phys. Today 62 (2009) 27–32, http://dx.doi.org/10.1063/1.3248476.
[34] R. D'Apolito, A. Perazzo, M. D'Antuono, V. Preziosi, G. Tomaiuolo, R. Miller, S. Guido, Measuring interfacial tension of emulsions in situ by microfluidics, Langmuir 34 (2018) 4991–4997, http://dx.doi.org/10.1021/acs.langmuir.8b00208.
[35] H.M. López, J. Gachelin, C. Douarche, H. Auradou, E. Clément, Turning bacteria suspensions into superfluids, Phys. Rev. Lett. 115 (2015), 028301, http://dx.doi.org/10.1103/PhysRevLett.115.028301.
[36] Hung-Chieh Wu, Lin-Chien Lee, Wei-Jie Wang, Plasmapheresis for hypertriglyceridemia: the association between blood viscosity and triglyceride clearance rate, J. Clin. Lab. Anal. 33 (2019) e22688, http://dx.doi.org/10.1002/jcla.22688.
[37] M. Ranucci, T. Laddomada, M. Ranucci, E. Baryshnikova, Blood viscosity during coagulation at different shear rates, Physiol. Rep. 2 (2014) e12065, http://dx.doi.org/10.14814/phy2.12065.

Biographies

Luca Lanotte is a Junior Scientist of the Institute of National Institute for Agricultural Research currently working at the laboratory of Science and Technology of Milk and Egg (STLO – Rennes, France). He obtained a Ph.D. in Physics and Chemical Engineering from the University Federico II of Naples (DICMAPI, Italy) and the University Joseph Fourier of Grenoble (LIPhy, France). The Ph.D. project dealt with the study of human microcirculation using a microfluidic and biomimetic approach. From 2013 to 2017, he was postdoctoral fellow at the Centre de Biochimie Structurale (CBS – Montpellier, France). He contributed to the development of projects focusing on the microfluidic and the rheology of blood and he provided a new look at red blood cell dynamics under high shear rates. Since September 2017, he joined the "Spray-drying - Concentrated Matrices - Functionalities (SMCF)" research team of the STLO laboratory in Rennes. The main goal of his research is to shed light on the mechanisms leading to milk drying using a multiscale approach, and to understand how the management of concentration/destabilization operations (evaporation-spray-drying/rehydration) affects the production and the properties of milk powders.

Giovanni Ausanio received his M. Sc. in Physics from "Federico II" University of Naples in 1997 and the Ph. D. in Materials Engineering from the same university in 2004. Apart the Ph. D. scholarship, after graduation he had a two years INFM (National Institute for the Physics of Matter) grant (1997-1999)(1997–1999) and a 16 months INFM contract of formation as technologist (2000–2001). Between 2004–2006 he was CNR-INFM TD researcher. Between 2006–2015 he was assistant professor and from 2015 he is associate professor at University of Naples "Federico II", Department of Physics. He is co-author of 100 publications in International Referred Journals, 5 reviews in books, 2 patents, 1 Encyclopedia contribute. He has attended different national and international research project. During his research activity, G.A. gained expertise in the fields of magnetic and magnetotransport properties in nanogranular films, and in the field of magnetoelasticity for the specific case of elastomagnetic composites materials. His experimental competences include: (a) morphological and magnetic domains characterisation of granular structures by atomic force microscopy (AFM) and magnetic force microscopy (MFM), respectively; (b) magnetic and magnetoresistive characterization using vibration sample magnetometry (VSM) and four-point probe method, respectively; (c) sensors/actuators prototypes for specific applications.

Vincenzo Iannotti received his Master in Physics from the University of Naples "Federico II" in 1994. From 1994 to 1995, he conducted research at the Research Unit of Naples of the National Institute for Physics of Matter (INFM) under grant INFM. He obtained his Ph.D. in Physics from the University of Naples "Federico II" in 1999. From 1999 to 2000, he was postdoctoral fellow at the INFM Research Unit of Naples and, from 2000 to 2002, he was INFM Researcher. From 2002 to 2016, he was University Researcher and Assistant Professor and, since 2016, he is associate professor of experimental physics at the Department of Physics "E. Pancini", University of Naples "Federico II". His research interests focus on nanostructured magnetic materials and their applications, with emphasis on magnetic nanoparticles and magnetic nanoparticle assembled films, and elastomagnetic composites for sensors. The results of his research activity are reported on about 80 papers published on international scientific journals, in five papers published on international scientific books and in two patents. He is also Member of the European Magnetism Association (EMA), the Italian Magnetism Association (AIMagn), the American Nano Society and the Italian Physical Society (SIF), and reviewer of several international journal in materials and physics field.

Giovanna Tomaiuolo is Assistant Professor in Transport Phenomena at the University of Naples Federico II. She received her PhD in 2009 working on the rheology of complex fluids as blood and droplet-based systems. Her main research interests concern fluid dynamics in microfluidic devices, microfabrication, rheology, microreactive flows, and structure-property relationships in complex biological fluids, emulsions and water-surfactant systems. Currently, she is working on surface functionalization to face fouling and clogging phenomena at the micro-scale.

Luciano Lanotte is full professor of Experimental Physics at the University of Naples "Federico II", Chief of the Magnetism Group in the Department of Physics of this University. He published more than 120 papers in the field of new magnetic materials modeling and application, and is recognized as expert in the topic of elastomagnetim, receiving numerous invitations for talks at international conferences. In particular, he was: Chairman of the 1 st MEA '93 "International Conference on Magnetoelastic Effects and Applications" Capri, 24–26 May 1993; Organizer of the Symposium "Magnetostrictive, magnetoelastic, multiferroic, ferromagnetic shape memory materials and application" for Joint European Magnetic Symposia, JEMS 06, San Sebastian (Spain) 26–30 June 2006; Organizer of the Symposium "Magnetic shape memory, magnetoelastic and multifunctional materials" for Joint European Magnetic Symposia, JEMS 12, September 9–14, 2012, Parma, Italy. He was awarded as valued reviewer in 2009, for the contribution given to the quality of Sensors and Actuators A.