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Inductively Coupled Resonant Humidity Monitoring Exploiting Irreversible State Changes

1. Auflage

Technische Universität Dresden

Inductively Coupled Resonant Humidity Monitoring Exploiting Irreversible State Changes

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von der Fakultät Elektrotechnik und Informationstechnik der
Technischen Universität Dresden

zur Erlangung des akademischen Grades eines

Doktoringenieurs
(Dr.-Ing.)

genehmigte Dissertation

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Tag der Einreichung:	09.10.2015
Tag der Verteidigung:	26.07.2016

Herstellung und Verlag:
BoD - Books on Demand, Norderstedt
ISBN 978-3-7431-6868-8

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Nomenclature

φ	relative humidity (r.H.) in $\%$
φ_D	deliquescence relative humidity in $\%$
φ_E	efflorescence relative humidity in $\%$
A	area in m^2
m	mass in kg
t	time in s
w_φ	humidity sorption rate, above the deliquescence relative humidity
X_D	index, typical detection
X_{IM}	index, imaginary
X_{lo}	index, lower boundary
X_{MAG}	index, magnitude
X_{PHA}	index, phase
X_{RE}	index, real
X_R	index, typical reference
X_S	index, typical sensor
X_{up}	index, upper boundary
ω_X	angular frequency with index $_X$ representing a specific frequency characteristic
ϵ'	electric permittivity real part / dielectric constant
ϵ''	electric permittivity imaginary part / dielectric loss
$\epsilon*$	complex electric permittivity
ϵ_0	vacuum electric permittivity
ϵ_r	static relative electric permittivity
ϵ	electric permittivity
$\hat{\rho}$	density in $\frac{\mathrm{kg}}{\mathrm{m}^3}$
μ''	magnetic permeability imaginary part
μ'	magnetic permeability real part
$\mu*$	complex magnetic permeability
μ_0	vacuum magnetic permeability
μ_r	static relative magnetic permeability
ϕ_B	magnetic flux
ρ_0	specific electrical resistance in Ωm
B_W	bandwidth in Hz
B	magnetic flux density in T
C_R	electric resonator capacitance in F
C_S	electric sensor capacitance in F
C	electric capacitance in F

D	electric displacment field/electric flux density in $\mathrm{C/m^2}$
E	electric field in $\frac{\mathrm{V}}{\mathrm{m}}$
$f_{0,Im}$	sensor imaginary part resonance frequency
$f_{0,Mag}$	sensor magnitude resonance frequency
$f_{0,Pha}$	sensor phase resonance frequency
$f_{0,Re}$	sensor real part resonance frequency
f_0	sensor resonance frequency
$f_{i,X}$	higher resonance of type X
f	frequency in $\mathrm{s^{-1}}$
H	magnetic field strength in $\frac{\mathrm{A}}{\mathrm{m}}$
I	electric current in A
L_R	electric reference resonator inductance in H
L_S	electric sensor resonator inductance in H
L	electric inductance in H
p	pressure p in Pa
Q	electric charge in C
R_S	sensor resistance in Ω
R	electric resistance in Ω
$S_{i,j}$	scattering parameter for input port i to output port j with i,j=1..2
s	complex angular frequency $s = \sigma + j\omega$
T	temperature in K
U	electric voltage in V
V	volume in $\mathrm{m^3}$
Y	electric admittance in Ω
Z	electric impedance in Ω
AC	alternating current
ADC	Analog-to-Digital-Converter
BAW	bulk acoustic wave
CRH	crystallization relative humidity
CTI	critical temperature indicators
D(F)FT	discrete (fast) Fourier transformation
DAC	Digital-to-Analog-Converter
DC	direct current
DDS	discrete direct synthesis technique for frequency generation
DRH	deliquescence relative humidity
DUT	device under test
EM	electromagnetic
ERH	efflorescence relative humidity
ESD	electrostatic discharge
FD	finite difference
FDM	finite difference method
FE	finite element

FEM	finite element method
FFT	fast Fourier transformation
FM	frequency modulation
FMS	frequency modulation spectroscopy
FPGA	field-programmable gate array
FRI	frequency range of interest
FV	finite volume
FVM	finite volume method
FWHM	full width at half maximum
GPIO	general purpose input output interface
HF	high frequency
HMI	humidity indicator card (typically reversible)
IC	integrated circuit
ICR	inductively coupled resonant (wireless passive sensor principle)
IHMI	irreversible HMI
IREV-C	proposed sensor principle based on an irreversible capacitive change
IREV-R	proposed sensor principle based on an irreversible resistance change
IREV-RMIX	irreversible resistance change sensor principle variant (intermixed NP region)
ITRS	International Technology Roadmap for Semiconductors
LPS	liquid phase sintering
MATPEN	Matrix Pencil method
MOD	metal organic decomposition complex (ink type)
MS	metallic salt (ink type)
NFC	near field communication
NP	nano particle
NWA	network analyzer
OSI	open systems interconnection model
PCB	printed circuit board
PPM	parts per million
PPMv	parts per million by volume fraction
PPMw	parts per million by molecular weight
REV	representative elementary volume
RFID	radio frequency identification
ROM	read only memory
RX	reception
SAW	surface acoustic wave
SDR	software defined radio
SEM	scanning electron microscope images
TTI	time temperature integrators
TX	transmission
UHF	ultra high frequency
VHF	very high frequency

Acknowledgments

As we are standing on the shoulders of giants, we make yesterday's science fiction today's reality, but on our way we should never forget to think before pushing buttons ... .

This study was conducted while I held the position of scientific assistant at the Institute of Semiconductors and Microsystems Technology (IHM), at the chair of Microsystems Technology of the Technical University Dresden. The study was partially funded by the DFG as part of the Collaborative Research Center SFB639 'Textile-reinforced composite components for function-integrating multimaterial design in complex lightweight applications', as well as by the BMBF with the project INHUEPRO - 'Intelligent Hip Prosthesis'.
My profound gratitude goes to Prof. Dr.-Ing. habil. Wolf-Joachim Fischer for taking on the responsibility of supervising and guiding this thesis and for providing the framework within which it could be created. I also would like to thank Prof. Dr.-Ing. Niels Modler for taking on the responsibility of reviewing this thesis.
Furthermore, my sincere appreciation goes to Dr.-Ing. habil. Uwe Marschner for "opening the door" to the position at the institute and encouraging me to publish the results of my work. I am grateful to colleagues Barbara Adolphi, Adelheid Chares, Andreas Kunadt, Maik Müller, Marion Schneider, Matthias Plötner for the cooperation. In addition, I very much appreciate to have had my colleague, Dr. Alexander Türke, introduce me to the mysterious and wonderful world of nanoscience and technology and assist me in rediscover chemistry and the various possibilities it offers.
Thanks also go to the former graduating diploma students Rene Körbitz, Ricardo Fiedler, Marcus Dehmel, Peter Weissbecker, and Sebastian Matthes for their project work related contributions. This applies equally to the study project thesis workers Martin Oelschlägel, Clemens Bartl, Pei Chen, Qian Wang, Yunbo Zhang, and students Marcel Gehringer and Clemens Todt.
Last but not least I would like to thank my family and especially Tati for their continuous support and Matheo for helping me readjust my point of view on life.

To those who passed on the way Elisabeth, Gerhard, Achim and, especially Axel

Abstract

Due to the pervasive nature of water, water vapor, and moisture, there is a strong influence on the product quality of a multitude of goods (e.g., food, chemicals, electronics, ammunition, etc.). Humidity as environmental water vapor is therefore of strong interest for the purposes of measurement and control throughout the life cycle of goods not only in regard to their use but also during manufacture, transport, and storage. One of the key requirements of monitoring measurement systems is to determine critical threshold or accumulated dosage exposure conditions. Nowadays, RFID technology is established, and a large number of standardized and non-standardized solutions of differing complexity exist. Sensor-enhanced RFID tags not only provide unique identification information but also additional sensor information. Fulfilling a monitoring task on item level is challenging when there is no continuous supply of electric energy available, a common application constraint in sensor-enhanced RFID applications. Application constraints are impeded due to the low cost requirements on the RFID market. Wireless passive humidity monitoring sensor solutions, in which the exceedance of a humidity threshold leads to a permanent, preferably irreversible change of a sensor parameter are proposed in this study. In the presented solutions, this is either a lasting electric resistance (IREV-R sensor approach) or an electric capacitance change (IREV-C sensor approach). For this purpose a number of physico-chemical phenomena are technically exploited in different sensor arrangements. These are the deliquescence of salts as threshold detection mechanism, transport processes in porous media as well as chemical liquid phase sintering of metal nanoparticles. The sensor principles introduced effectively act as humidity threshold-activated relative humidity dosimeters. For use in combination with RFID tags single use, low-cost sensor solutions are favored. Inkjet print as a representative mass production technique for printed electronics is examined in more depth, and its application exemplified for the IREV-R sensor principle. The suitability of the proposed irreversible principles for use in wireless passive monitoring sensor solutions is demonstrated via integration in inductively coupled resonant (ICR) sensor tags. This non-tactile measurement principle is another major field in the presented study. Aspects covered are the ICR sensor tags themselves, a treatment of the electric ICR system in the time and frequency domain as well as resonance frequency time domain signal analysis techniques. Due to its good performance in comparison to a large number of other signal analysis techniques the Matrix Pencil Method was investigated in more detail. The utilization of a double planar sensor coil arrangement as an unifying ICR sensor platform for the IREV-C and IREV-R approach is proposed. Theoretical, numerical and laboratory experimental results, which demonstrate the feasibility of the proposed sensor principles and developed solutions, are presented.

Kurzfassung

Die Funktion, Qualität und Lebensdauer von Gütern wird maßgeblich durch die auf sie einwirkenden Umgebungsbedingungen beeinflusst. Neben der Temperatur ist die relative Luftfeuchte ein kritischer Umgebungsparameter von hoher Bedeutung für z.B. Lebensmittel, Chemikalien, Medikamente, elektronische Produkte oder militärische Güter. Eine Überwachung der wirksamen relativen Luftfeuchte während des Produktlebenszykluses, umfassend Herstellung, Transport, Lagerung und Einsatz ist daher von besonderem Interesse. Eine typische Aufgabe überwachender Messsysteme ist es Grenzwertüberschreitungen oder kritische, akkumulierte Dosen zu erfassen. Weiterhin ist die RFID Technik gegenwärtig etabliert und es existieren verschiedene standardisierte und nicht- standardisierte Verfahren unterschiedlicher Komplexität zur Produktidentifizierung oder angeschlossenen Aufgabenstellungen, wie der Authentifizierung. Bekannt sind auch vielfältige Varianten sensorischer RFID Tags, welche nicht nur Identinformation, sondern auch zusätzliche Sensorinformationen bereitstellen. Soll mit diesen eine Überwachung kritischer Umgebungsparameter auf Ebene des individuellen Produkts während seines Lebenszykluses möglich sein, ergeben sich nicht nur besondere Herausforderungen hinsichtlich einer kontinuierlichen, elektrischen Energieversorgung, sondern auch hinsichtlich eines herrschenden, extremen Kostendrucks im Bereich der RFID Technik. Im Rahmen dieser Arbeit werden passive, drahtlose, relative Luftfeuchte überwachende Sensorlösungen vorgestellt, in denen Schwellwert überschreitende, akkumulierende Dosen zu permanenten Änderungen elektrischer Sensorparameter führen. Im Speziellen entweder zu einer dauerhaften Änderung des elektrischen Widerstandes (IREV-R Sensoransatz) oder der elektrischen Kapazität (IREV-C Sensoransatz). Zu diesem Zwecke werden verschiedene physikalisch-chemische Phänomene technisch in Sensoranordnungen ausgenutzt. Dies umfasst das Deliqueszensverhalten von Salzen, Transportprozesse in porösen Medien, sowie chemisches Sintern von Metall- Nanopartikeln in flüssiger Phase. Die vorgestellten Sensorprinzipien und abgeleiteten drahtlosen Sensoren arbeiten als relative Luftfeuchtegrenzwert aktivierte Dosimeter. Da diese insbesondere zum Einsatz in RFID-Tags geeignet sein sollen, sind Einmalsensoren, mit vergleichsweise geringen Herstellungskosten bevorzugt. Insbesondere wird der Inkjetdruck, als eine mögliche Fertigungstechnologie gedruckter Elektronik vertieft betrachtet. Dessen Anwendung zur Herstellung wird am Sensorprinzip mit dauerhafter, elektrischer Zustandsänderung präsentiert. Die generelle Eignung der vorgeschlagenen Sensorprinzipien für überwachende, drahtlose, passive Sensorsysteme wird am Beispiel induktiv, gekoppelter, resonanter (ICR) Sensoren gezeigt, auf deren Grundlage Sensoranordnungen entwickelt und gezeigt werden. Bei diesem Messprinzip handelt es sich um ein weiteres Kernarbeitsthema dieser Arbeit. Behandelte Aspekte umfassen hierbei deren analytische Behandlung im Zeit- und im Frequenzbereich, Zeitbereichssignalanalyseverfahren zur Resonanzfrequenzextraktion im Allgemein, sowie die Matrix Pencil Methode im Speziellen. Eine Doppelplanarsensorspulenanordnung für das IREV-R und IREV-C Messprinzip wird vorgeschlagen. Präsentiert werden theoretische, numerische und experimentelle Ergebnisse, welche die Anwendbarkeit der vorgeschlagenen Lösungen verdeutlichen.

1 Introduction

1.1 Towards Completely Printed Sensor-enhanced RFID Tags

RFID[1] tags permit the identification and surveillance of individual items or item groups. One of the self-declared goals of the tag manufacturing industry is the replacement, or at least an cost-effective alternative, to the ubiquitous optic barcode with an electronic counterpart. Typical stated advantages [162, 78] of such an electronic barcode are an increased read range and data depth, non line-of-sight communication not necessarily only through air but also through non-opaque item package media, higher robustness against pollution, and/or the possibility to detect multiple RFID tag signatures during a single read process with a single reader device. One reason why RFID tags have not replaced the optical barcode lies in their manufacturing costs, which are still higher (currently ranging between $ 0.10 to 0.15[2]). Therefore, in general, RFID tags are not yet competitive when compared to the optic barcode (costs between $ 0.02 to 0.05) [163, 164]. Nevertheless, the use of RFID tags constantly increases, especially in application fields in which their benefits outweigh the price drawback. Prominent examples can be found in the sectors of supply chain management, asset tracking, security access control, animal identification, vehicle identification in toll collect systems, item tracking in libraries, or in high valued consumer products (cosmetics, pharmaceuticals, clothing), to name only a few.

silicon IC based RFID tags (A)		all printed chipless RFID tags (B)	
with external peripheries (A1), using a mix of components and manufacturing strategies, (i.e., silicon RFID IC and screen printed antenna)	everything on chip (A2), with all components integrated, including the antenna (i.e., smart dust)	transistor-based with logic circuits (B1), all RFID tag's elements are manufactured via printing technologies (in development)	transistor-less without logic circuits (B2), alternative RFID tag approaches and read-out principles are used (in development)

Table 1.1: Possible RFID tag development routes.

Different development trends can be distinguished, all of them aiming at a cost reduction of the individual RFID tag (Tab.1.1). In the classic development routes (A1, A2), ICs are manufactured via established semiconductor production technologies, and a cost reduction is reached due to a size reduction based on the integrated circuit (IC) scaling laws as, i.e., manifested in the ITRS[3] road-map (Moore and More than Moore [92]). A major manufacturing bottleneck for RFID tags is hereby currently required interconnects to external components such as antennas or, if used additional components such as sensors or batteries, since the RFID ICs must still be processable in the subsequent assembly processes [177]. If this bottle-

[1] radio frequency identification
[2] large quantities of passive IC based tags with substrate mounted chip and printed antenna
[3] International technology road map for semi-conductors

neck is eliminated, e.g., via an antenna integration directly into the IC (A2), for which a prerequisite is an increase in the operation frequency, scaling via miniaturization will again become feasible [96, 165]. A second evolving development route emerges with the field of printed electronics. A cost reduction is reached by an increase in the throughput due to a high manufacturing speed and a strong increase in the processable area, a reduced number of processing steps, and the possibility to use low cost substrates. Printed electronics rely on digital printing techniques suitable for a role-to-role production such as inkjet or offset print. Besides printing the RFID tag component with the largest size requirement, the antenna[4] a research aim in this field, is to completely print RFID tags (including logic). Two development directions can be distinguished: transistor-based RFID tags (B1) and transistor-less RFID tags (B2). Most likely all development directions will find their specific application fields, and emerging RFID devices such as the transistor-less RFID tags will not replace the traditional silicon based electronics but will rather be a complement with unique characteristics able to provide application-specific advantages. Having available an infrastructure, a market, manufacturing technologies, standards, and research as well as development potential, a logic consequence for RFID tags is to not only supply identification information but also additional sensor information (ubiquitous sensing). These sensor-enhanced RFID tags (i.e., [216, 82]) can be used to monitor critical environmental parameters acting on item level. For this purpose, a supply of electric energy, an additional data storage mechanism (either on-tag with writable memory or off-tag in the reader), and signal processing capabilities are required when established sensor principles and sensors are employed. This leads to the requirement of using either semi-passive[5] or active RFID tags with an additional battery[6], or in the case of passive tags, one or more required omnipresent reader stations. An alternate solution approach pursued in this study relies on 'unconventional' transducer principles based on an irreversible change of a transducer parameter which is caused by the environmental parameter of interest (of high relevance are temperature and relative humidity, closely followed by acceleration and tilt). Recent developments in the material sciences (nanotechnology research) have led to materials which electrical properties can change significantly and permanently in dependence on the environmental conditions. As demonstrated in this study, this permits continuous environmental parameter monitoring with passive RFID tags without the requirement of an additional electric energy source and a writable data storage. In addition, this approach may be the only option for upcoming transistor-less RFID tags without integrated logic circuits (i.e., presented in [163]). Besides temperature, relative humidity is most likely the second most important environmental influence with critical impact on product quality as discussed briefly in the following chapter.

1.2 Humidity Impact and Promises of Lowest Cost Monitoring Solutions

Humidity[7] affects air properties and the properties of materials in contact with air. As remarked by Fenner and Zdankiewicz [48]: 'water is the most pervasive chemical on our planet with an impact on a plurality of applications ranging from weather forecast, human comfort to product quality and safety to

[4] for frequencies below 2.4 to 4.8 GHz
[5] interchangeably semi-active
[6] or possibly an energy harvester (excluding the employed frequency band(s) of the electromagnetic spectra used by the RFID tag to communicate with a reader)
[7] which is the water content in gases, whereas moisture is the water content in solids

application field	r.H. values/range	possible effects (<..below / >..above threshold)
residential indoor comfort	30..70 / 50..60	increased sweating (>) / mucosa drying out (<)
manufacturing		
- electronics	30..70	ESD[a] (<), conductive anodic filament failure (>)
- semiconductors (clean room)	36..39 / 30..50	electrostatic charging (<)
- food processing (food dehydration)	0..50	early spoilage, bacteria and mold growth(<)
- printing (paper)	40..55	warping (>)
- ceramics drying	0..50	mold growth (>)
- chemicals (agriculture)	differing	caking, bleaching (>)
medical/hospital		
- operating room	50..60	sterilization, bacteria growth (>)
- infant incubator	50..80	permanent health hazards (<,>)
museums and galleries		
- Smithsonian (general)	37..53	object damage (<,>)
– material: woods	30..62	object damage (<,>) (fully restrained, no initial stress)
– material: woods	30..80	object damage (<,>) (fully restrained, with initial stress)
– material: titanium paint	28..66	object damage (<,>) (fully restrained, no initial stress)
– material: earth color paint	30..64	object damage (<,>) (fully restrained, no initial stress)
electronic packaging standard		
- IPC/JEDEC JEDEC 033A	5 / 10 / 15	-
- IPC/JEDEC JEDEC 033B	5 / 10 / 60	-
military standard (U.S.)		
- MS 20003-2	40 / 40 / 50	-
- MIL-I-8835	30 / 40 / 50	-

[a] electrostatic discharge

Table 1.2: Examples of relevant relative humidity ranges for different application fields [48, 124].

window defoggers in cars', to name only a few examples from the various affected processes. In comparison to other environmental quantities with an impact on product quality (i.e., function, appearance), safety and life time, the number of humidity-caused defects and failures closely follows temperature-related ones. Due to the relevance of water vapor and moisture to, e.g., chemicals, building materials, pharmaceuticals, fertilizers, electronics, food and cosmetics, measurement and monitoring is carried out whenever there is a need to detect and/or prevent, e.g., condensation, corrosion mold, or warping. Examples of applications, fields, and related tolerable humidity ranges as well as possible failure modes are listed in Tab.1.2. Besides item tracking, the mentioned sensoric RFID tags used for measurement, monitoring and control purposes can increase product life time, and ensure product safety as well as functionality. As outlined by Unander [205] such an approach leads to several requirements, which the concept of a sensor-enhanced RFID tag should satisfy. It should ideally provide a long life time (I), and be of very low cost (II), while it should be possible to hide or incorporate the device inside objects (III), i.e., in the product wrapping. A current state-of-the art monitoring system would be an active or semi-passive RFID system with integrated batteries and either a commercial or a customized sensor that continuously logs humidity and can be queried to report threshold violations (i.e., [82]). This would satisfy condition (III) but it has a limited life time (I) because of the finite integrated energy source. In dependence on the integrated components, this approach would be of high or medium costs (II). In opposition known optical indicator cards (see Sec.2.3), which visually indicate an exposure to harmful conditions, currently satisfy the low cost (II) and long lifetime criteria (I) easily, yet an integration in product enclosures is not generally possible due to the requirements of a visual readout and transparent packaging media (III). One aim of this study are wireless humidity monitoring solutions with the ability to fulfill all of the mentioned requirements.

2 State of the Art

In the following, an overview of humidity measurement principles, with a focus of miniaturized sensors and miniaturizeable measurement principles, wireless passive sensors and sensor systems as well as measurement approaches exploiting irreversibility for sensing purposes is presented. This chapter provides the technical context for the presented study from a system engineer's point of view. In the overview of the field of wireless passive sensors an emphasis is on solutions of the lowest electric complexity, meaning IC-less or transistor-less wireless passive sensor solutions, especially inductively coupled resonant sensors, as they are used in this study as a monitoring wireless sensor tag demonstration platform.

2.1 Humidity Measurement Principles and Sensors

Humidity is the amount of water vapor[1] in a gas volume, in this study, air. It can be quantified as either absolute or relative humidity (additional expressions such as specific humidity or dew point exist). Absolute humidity is an expression for the total amount of water vapor present in a unit volume of air (i.e., in $\frac{\mathrm{g}}{\mathrm{cm}^3}$ or $\frac{\mathrm{kg}}{\mathrm{m}^3}$, PPM[2] or D/F pt[3]) and has extensive applications in industry especially in trace moisture measurement. For the determination of environmental (atmospheric) water vapor content commonly the relative humidity φ (or r.H. in $\%$ or unitless) is of interest since a water vapor interaction with a material is often in proportion to this quantity or starts when a specific activation threshold is exceeded. It is defined as the ratio of the partial pressure of water vapor p_w present in a gas to the saturated vapor pressure p_s of the gas at a temperature T_0 (Eq.2.1) [168].

$$\varphi\Big|_{T=T_0} = \frac{p_w}{p_s} \cdot 100\,\% \qquad (2.1)$$

Humidity measurement carried out with hygrometers belongs to the more difficult problems in basic metrology. As stated by Rittersma [168]: hygrometry is 'a branch of applied physics, in which the multitude of techniques is an indication of the complexity of the problem, and of the fact that no one solution will meet all requirements at all times and in all places'. Due to the various interaction mechanisms of water with other materials, a broad range of humidity measurement principles exist, whereby the majority of the approaches rely on measurements of one or more humidity-dependent quantities and the absolute or relative humidity is obtained indirectly via additional calculations. A varying water vapor content in air surrounding a measurement device can lead to a change in temperature, electric impedance, pressure, length and mass. Water absorbs infrared and ultraviolet light and changes the color and crystal structure of chemicals (hydrate formation), the refractive index of air and liquids, the velocity of sound in air or electromagnetic radiation in solids and the thermal conductivity of gases as well as that of liquids and solids. Observable changes can be the result of water adsorption, absorption, evaporation or

[1] not to be mistaken for steam
[2] either in PPMv parts per million by volume fraction or PPMw by molecular weight
[3] dew/frost point