# Scientific Report ESF Workshop 04-109

**Title:** **RARE EARTH OXIDE THIN FILMS: growth, characterization, and applications**

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## 1. Executive summary

The Workshop took place in Sanremo from Wednesday May 11 to Friday May 13 2005, at the Villa Nobel in Sanremo (Imperia) - Italy. The Villa Nobel is the place Alfred Nobel used as home and laboratory during his stay in Italy. The Workshop was included in the list of events the Nobel Foundation organized to acknowledge the World Year of Physics 2005. Twenty nine people attended the meeting: the two Convenors, an ESF/PESC representative, 21 invetees, and 5 researchers of the MDM-CNR/INFM National Laboratory (the Convenor affiliation). The 21 invetees were chosen among the most prominent european and world-wide acknowledged scientists dealing with rare earth (RE) based compounds from both industry and academia.

The main goal of the Workshop was to discuss thin RE oxide films as emerging materials for microelectronic, nanoelectronic, spintronic, and optoelectronic applications. Twenty four invited presentations, 25 minutes long, each followed by a 5 minute discussion, aimed at illustrating the state-of-the-art of thin film deposition techniques, as well as the structural, physical, chemical, and electrical properties of thin RE oxide films and of their interface with the substrates on which ther were deposited. The goal was to identify proper methodologies for the development of RE oxide films, and to evaluate their effectiveness as innovative materials in the various above mentioned applications.

A total of 8 sessions were organized. Sessions 1, 2, and 3 were devoted to chemical and physical deposition methods of RE oxide films. Among the chemical deposition methods, the invited specialists introduced atomic layer deposition (ALD), and metal-organic chemical vapor deposition (MOCVD). Among the physical deposition techniques, molecular beam epitaxy (MBE), and pulsed laser deposition (PLD) were presented. Growth mechanisms and precursor chemistry (especially for ALD and MOCVD) were examined both from the theoretical and the experimental point of view. Basic structural, morphological, compositional and electrical characterization of the thin RE oxide films was discussed.

Session 4 focussed on the structural and compositional characterization of RE oxide films, with emphasis on film and interfacial layers, on local structure and on electronic properties. All these factors play a key role in establishing the effectiveness of RE oxide films in advanced device applications. These characteristics were examined with advanced laboratory techniques (such as Rutherford back-scattering - RBS, low energy ion - LEIS, and x-ray photoelectron – XPS spectroscopies), with synchrotron radiation x-ray absorption based methods (such as extended and near edge fine structures - EXAFS and XANES), and finally with ultramicroscopy techniques (such as conventional and high resolution electron microscopy - TEM and HREM, conventional and valence electron energy loss spectroscopies – EELS and VEELS, and energy loss near edge fine structure - ELNES). The effort described in this session indicates how difficult it is to investigate interfacial layers.

A thorough review of the elecrical properties of RE oxide films and of their interfaces with various low or high mobility semiconductors was offered in session 5. In the latter, measurement and origin of the dielectric constant ($\kappa$) value, formation of fixed charges, and origin of the interfacial defect states, their dependence upon semiconductor substrate type and orientation, their modifications upon annealing, were discussed for RE oxide films. Session 6 examined both experimentally and theoretically the band alignment of RE oxide films embedded between a metal gate and various low or high mobility semiconductors in metal-oxide-semiconductor (MOS) devices. Measurements of band gaps ($E_g$) were also discussed. Band alignment, usually measured using internal photoemission spectroscopy (IPE) and XPS, plays a key-role in determining high-$\kappa$ oxide efficiency in real devices.

Sessions 7 and 8 were devoted to the applications of RE oxide films. In session 7, the micro- and nanoelectronic applications (in particular in ultra-scaled complementary MOS devices) were examined. The challenge of device downsizing was presented, in particular the reduction of the channel length below the 32 nm node (15 nm channel lenght) expected within 2008, and correlated with the search of suitable high-κ oxides. The latter were supposed offer an equivalent dielectric gate oxide thickness of about 0.8 nm at a physical thickness of more than 2 nm, thus limiting considerably leakage currents. Candidate κ values must range between values higher than the one of $SiO_2$ (3.9) but lower than those giving rise to significant short channel effects (about 45). RE oxides fall within this interval. In session 8, further microelectronic, spintronic and optoelectronic applications were discussed. RE based compounds such as $REMnO_3$ are of interest in non-volatile memory devices because of their magnetoferroelectric properties. In spintronics, interest toward RE-based compounds arises because they can promote giant or colossal magnetoresistive effects in spin valves (e.g. through $RE_xSr_yMn_zO_t$ type of compounds) for spin-light emitting diodes, -field effect transistors, and -qubits for quantum computers. Moreover, the half-metallic properties of these complex oxides make them appealing for their use as ferromagnetic-metallic layers in spin valves. RE oxides are also proposed in optoelectronics as laser host materials and as active waveguide structures to be integrated in chip technology. In the former case, RE oxides can give rise to a large intensity versus length product at low laser threshold.

Two special discussions took place during the last day of the Workshop. A scheme of the discussion was offered, and all participants contribuited to the discussion according to the proposed themes. The first discussion focussed on the relationship between microstructural, electrical, and electronic properties of RE oxide films. Unresolved issues were discussed (e.g. precise determination of RE oxide film κ, interfacial layer thickness and composition, band alignment between RE oxides and semiconductors using various techniques, the role of effective mass and of the *f* shell filling level in the RE elements, the relationship between defects and mobility, and the effects of annealing on the κ value). The second discussion focussed on the applications of thin RE oxide films and of their perspectives. The future of RE oxides on high mobility and high power semiconductors, the magnetoelectric and magnetocaloric effects in some RE-based compounds, the critical role of interfacial layers in ferromagnetic tunnel junctions, and the engineering of RE based compounds were included in the discussion.

In conclusion, presentations and discussions showed that RE oxides are of real interest for microelectronic, nanoelectronic, spintronic, and optoelectronic applications, and that a number of issues related to basic scientific and technological problems still need to be addressed. Collaboration among the highly qualified participants to the Workshop was suggested to be advantageous to fulfill this challenging task.

## 2. Scientific content of the event

Six main themes emerged during the 8 sessions of the Workshop: precursors for ALD and MOCVD, crystalline structure engineering, interfacial layer structure, electrical properties and band alignment on semiconductors, and finally applications of RE oxide films. These themes are here after briefly summarized and commented.

**• Precursors for ALD and MOCVD.**

Precursor quality is the fundamental resource to produce good quality RE oxide or RE-based compound films through chemical deposition methods such as ALD and MOCVD. Based on the requirements for suitable precursors for these techniques (i.e. volatility, no self-decomposition below the deposition temperature, sufficient reactivity, no eching of the substrate or of the growing film material), the following ligands were singled out up to date: "first" and "second generation" β-diketonates (e.g. thd = 2,2,6,6,-3,5-heptanedione, and $(\beta\text{-diket})_n$.poliethers respectively), ketoiminates, cyclopentadienyls (from the simple $(C_5H_5)_3$ to the largely discussed bis-cyclopentadieyl - as the $[(\eta^5\text{-}C_5H_4SiMe_3)_2LuCl]_2$ complex), alkoxides [e.g the promising methoxy-methyl-propanolate $O(CH_3)_2OCH_3$-based complexes], trimethylsilylamido- and aminidato type of complexes (which however do not generate self limiting growth - at least in ALD - because of the very limited precursor thermal stability), and finally di-$^t$butylphenolates. The RE ionic radius was acknowledged to affect the precursor performaces (e.g. the growth rate). In addition, type of ligand was found to determine the film resistance against degradation upon annealing. Improvement of precursor performances, and film growth challenge current research in RE precursor synthesis, and modeling of the growth mechanisms.

**• Crystalline structure engineering**

The film crystalline structure plays a significant role in determining the properties of both as grown and annealed RE oxide films. In particular, for applications of RE oxides as high-κ insulators in microelectronic devices it is necessary to increase as much as possible their crystallization temperatures to avoid irreversible film modifications and changes in the interfacial layer (IL) between the RE oxide film and the semiconductor upon annealing. Such modifications and changes could limit the performances of high-κ based devices (e.g degrade the electrical properties). Films grown epitaxially should in principle be less prone to structural changes upon annealing, and therefore, they should be the appropriate choice. For this reason research focussed on epitaxially grown RE oxides and RE-based compounds deposited using MBE, and PLD on various semiconductors and orientations. Phase stability and increase of order degree upon annealing were assessed. In addition, MBE capability in atomic level engineering and interface control of the deposited films are exploited to obtain well performing films. Ternary amorphous films could also turn out to be promising: crystallization temperatures even above 1000 °C were indeed found, together with good electrical properties in terms of κ values, band alignments, and leakage currents.

**• Interfacial layer structure**

Thickness and composition of the IL between high-κ oxides and semiconductor substrates affect many properties of MOS devices. In first place the κ value of the whole dielectric stack, and therefore the ultimate equivalent oxide thickness (EOT); secondly the interfacial defects. Therefore, it is crucial to precisely determine the IL characterstics. Most of such investigations were performed on films deposited on Si. It was established that annealing in whatever atmosphere promotes IL formation, even when a sharp interface was detected in as grown films. Moreover, the

IL composition was usually found to consist in $SiO_2$ and RE silicates, or a mixture of the two. IL consisting of RE silicides were detected in films deposited in ultra high vacuum (UHV), e.g. by MBE and PLD. In some cases, annealing might also promote changes in the oxidation state of the RE element. The presence of $Si^{2+}$ suboxides was correlated to strain at $Si/SiO_2$ interfaces, and the corresponding XPS peak area is used to determine the amount of interfacial trapping sites. Moreover, at $Si/SiO_2$ interfaces, annealing in inert ambient is shown to self-organize such Si-suboxide distribution and to lower the amount of interfacial defects at least. IL chemistry and thermodynamics on substrates other than Si are apparently different. Interestingly, in some cases no IL at all was observed (e.g. in ALD deposited films on Ge and GaAs using $H_2O$ as oxygen source).

**• Electrical properties of RE oxide films on semiconductors**

The κ value and the fixed charges in the dielectric layer, and the density of interface states ($D_{it}$) at the high-κ dielectric/semiconductor interface in MIS devices, determine profoundly the performance of micro-, nanoelectronic, and spintronic devices. **κ** values in RE oxide and RE-based compound were shown to range in a large interval (between about 10 to 30), and to depend upon many factors. e.g. composition, crystalline structure, and annealing treatment. In particular, the dependence of κ on dipole-carrying vibrational modes, which are detectable through infrared spectroscopy, was clarified. The vibrational modes are related to the crystal structure of the oxides, and, for RE oxides, those with cubic phase were shown to exhibit the absorption band with the highest effective charge at higher frequencies than those with hexagonal phase. This condition should induce for the former lower κ values than for the latter. **Fixed charge** formation was related to oxygen or RE interstitials and vacancies, to the readyness of RE oxides in absorbing moisture, and to Si contaminations in RE oxides on Si. Moisture was also shown to affect leakage current. Fixed charge reduction is generally expected after annealing. However, because of the existence in the phase diagrams of RE-compound phases with melting points congruent to trivalent RE silicates (which readily form at the IL), no interfacial relaxations at the RE silicate or oxide interfaces with $SiO_2$ are expected to occurr at temperatures above 800 °C. The opposite behaviour occurs in the case of $HfO_2$ films. As a consequence, no fixed charge reduction is predicted upon annealing of RE-based compounds above 800 °C. Finally, growth technique, precursor combination, and substrate were shown to affect $\mathbf{D_{it}}$. For example on Ge and GaAs, RE oxide films (the actual measurements were shown in particular for Lu and Yb oxides) deposited by ALD using $H_2O$ and $O_3$ as oxygen sources, exhibit lower $D_{it}$ values than those of $HfO_2$ films. This result was correlated with the RE to O stoichiometry ratio in the $RE_2O_3$ oxides, which promotes a lower number of oxygen atoms per RE one than in $TMO_2$ oxides – where TM indicates transition metal - (as, e.g., $HfO_2$). Moreover on Ge and GaAs, for RE oxide films lower $D_{it}$ values are generally measured than for those on Si.

**• Band alignment of RE oxides on semiconductors**

On a variety of RE oxides or RE-based compounds on various semiconductors (e.g. Si, Ge, and GaAs), IPE and XPS almost unanimously indicated a conduction band offset (CBO) between 2.0-2.2 eV in many of the candidate binary and ternary high-κ oxides, including also those containing RE elements. Not always theoretical predictions were shown to match the experimental results, and the CBO value almost fixed around 2.1 eV on various semiconductors is not explained yet. Moreover, not for the oxides of all RE elements the CBO and valence band offset (VBO) values were determined, either experimentally or theoretically. Interfacial layer chemistry was proposed also as a factor affecting band offsets. The role of the RE *f* shell filling level on the CBO value, however, is never discussed. In addition, in thin films with nanocrystalline structure, Jahn-Teller *d*-term splittings, states localized at grain boundaries between nanocrystals, and oxygen atom vacancies, determine defects in the band gap. These defects are supposed to affect the CBO values

measured in MOS devices. Finally, the discussion on band gap states is assumed to hold equally well for TM and for RE oxides. However, while in the former case theory and experiment are already at a mature stage, in the latter most of the work still needs to be carried out.

• **Applications**

**Micro- and nanoelectronics**: generally results of investigations on RE oxides for applications in micro- and nanoelectronic devices appear promising, although a number of issues must be addressed. Hygroscopicity affects negatively the electrical properties, however annealing can significantly improve them. The existence of an IL between the RE oxide and the semiconductor substrate might also be beneficial. Indeed it lowers the overall κ of the dielectric stack, but it also contributes to minimize mobility reduction inherently connected to remote phonon scattering. The choice of gate electrode in real MOS devices might contribute in improving the IL properties. Amorphous oxides seem to work better for real devices. Ternary compounds seem also to be very promising. Efficient non-volatile memory devices are achieved as both electric- and magnetic-fields can be used to switch the corresponding polarizations in a materials, such as $REMnO_3$. In this type of compound, the filled valence shells of the RE atoms shield the magnetism potentially present in them due to the unfilled 4*f* orbitals. This characteristics is one of the factors leading to magnetoferroelectricity. **Spintronics**: complex quaternary RE-based oxides deposited using ALD were shown to exhibit variable electronic resistivity values in a magnetic field on various substrates. This result suggests that these ALD deposited films could be applied in spin valves exploiting colossal magnetoresistive effects. If pursued, this achievement could enlarge the field of application of ALD films to spintronics. **Optoelectronics**: in this field, RE oxides are considered because of their superior thermomechanical properties, their strong Stark-splitting, and their low phonon energies. The former property makes them promising for high-power lasers, the second one enables efficient laser action, and the last one ensures large energy storage times.

## 3. Assessment of the results, contribution to the future direction of the field, outcome

Some of the work presented during the Workshop was carried out by already existing collaborations. Various collaboration groups could be identified, for example group 1 (Fanciulli, Zenkevich, Boscherini, Fiorentini, Schamm, Elliott, Dimoulas, Seguini, and Scarel), group 2 (Lucovsky and Schubert), group 3 (Palstra and Nilsen), and group 4 (Osten and Dąbrowski). These collaborations are occurring within completed, in progress, or just approved National, International, and European Projects. The opportunity to start new collaborations, or to enlarge the existing ones, emerged clearly. For example: between the theoreticians (Fiorentini and Dąbrowski), among groups interested in studying growth models and mechanisms, and precursors (Niinistö, Elliott, Haukka, Aspinall, and Scarel), among groups interested in electrical properties and band alignment (Fanciulli, Lucovsky, Robertson, Seguini), and among european groups interested and involved in device fabrication (Dimoulas, Fanciulli, Bär). To favour such collaborations, the participants expressed their desire to make the interaction stronger pursuing also the ESF **network** activities, possibly extended also towards the rest of the world, and organizing special events to discuss critical issues. A point that was particularly stressed was the promotion of beneficial interactions among different communities: microelectronics, magneto-electronics, and optoelectronics. Furthermore, most of the participants accepted to contribute to a book for the Springer-Verlag Series "***Topics in Applied Physics***" and entitled "*RARE EARTH OXIDE THIN FILMS: growth, characterization, and applications*" proposed by the Convenors. During the Workshop, guidelines for book chapter preparation were supplied.

## 4. Final programme

**Wednesday May 11, 2005**

13:15 – 14:15 Registration
14:15 – 14:25 Opening and Welcome (**M. Fanciulli**)
14:25 – 14:35 Presentation of the *European Science Foundation* (**G. Crean**, *Standing Committee for Physical and Engineering Sciences*)
14:35 – 14:45 Greetings of the President of the Imperia Province

**Introduction**
14:45 – 15:15 **G. Scarel** "*Scientific and technological issues of rare earth oxides: introduction to the main topics of interest of the Workshop*"

**Chemical deposition methods of rare earth oxides - I**
(Chair: S. Haukka)
15:15 – 15:45 **L. Niinistö** "*Atomic layer deposition of rare earth oxides*"
15:45 – 16:15 **S. D. Elliott** "*Models for ALD and MOCVD growth of rare earth oxides*"
16:15 – 16:45 **R. Lo Nigro** "*MOCVD of rare earth oxides*"

16:45 – 17:15 Break

**Chemical deposition methods of rare earth oxides – II** (Chair: L. Niinistö)
17:15 – 17:45 **S. Haukka** "*Experimental study of ALD growth mechanisms (with examples on rare earth oxides)*"
17:45 – 18:15 **H. C. Aspinall** "*Requirements of rare earth precursors for MOCVD (and ALD)*"

20:00 Dinner at the Royal Hotel - Sanremo

**Thursday May 12, 2005**

9:00 – 9:30 Guidelines for the Book Chapters for the Springer-Verlag Series "***Topics in Applied Physics***" (**M. Fanciulli**)

**Physical deposition methods of rare earth oxides** (Chair: A. Dimoulas)
9:30 – 10:00 **H. J. Osten** "*MBE of rare earth oxides*"
10:00 – 10:30 **J. Schubert** "*Fabrication and characterization of rare earth scandate thin films prepared by pulsed laser deposition (PLD)*"

10:30 – 11:00 Break

**Structural and compositional characterization of rare earth oxide films** (Chair: H. Iwai)
11:00 – 11:30 **A. Zenkevich** "*Film and interface layer composition of rare earth (Lu, Yb) oxides deposited by ALD*"
11:30 – 12:00 **F. Boscherini** "*Local structure of rare-earth oxides by x-ray absorption spectroscopy*"
12:00 – 12:30 **S. Schamm** "*Local structure, composition and electronic properties of rare earth oxides studied using ultra-microscopy techniques (TEM-EELS)*"

13:00 – 14:30 Lunch

**Electrical properties of rare earth oxide films and of their interfaces with semiconductors** (Chair: G. Bersuker)

14:00 – 14:30 **G. Lucovsky** "*Strain-relief at internal dielectric interfaces between* $SiO_2$ *and alternative gate dielectrics*"

14:30 – 15:00 **M. Fanciulli** "*Electrical characterization of rare earth oxides and of their interfaces with semiconductors* "

15:00 – 15:30 **V. Fiorentini** "*Theory of (and some experiments on) the dielectric properties of "high"-*$\kappa$ *La and Lu oxides*"

15:30 – 16:00 **J. Dąbrowski** "*Pseudopotential studies of rare earth oxides*"

16:00 – 16:30 Break

**Band alignment of rare earth oxides on semiconductors** (Chair: M. Fanciulli)

16:30 – 17:00 **G. Seguini** "*Experimental determination of the band offsets of rare earth oxides on different semiconductors*"

17:00 – 17:30 **G. Lucovsky** "*Conduction band edge states in nano-crystalline and non-crystalline high-*$\kappa$ *dielectrics*"

17:30 – 18:00 **J. Robertson** "*Band offsets of lanthanide gate oxides*"

20:00 Dinner at the Braccioforte Restaurant - Imperia

**Friday May 13, 2005**

**Special discussion**

9:00 – 10:00 "*Relationship between microstructural, electrical, and electronic properties of rare earth oxides"*. Coordinators: G. Lucovsky and F. Boscherini

10:00 – 10:30 Break

**Applications I** (Chair: G. Lucovsky)

10:30 – 11:00 **H. Iwai** "*Rare earth oxides in microelectronics*"

11:00 – 11:30 **G. Bersuker** "*Requirements of oxides as gate dielectrics in CMOS devices*"

11:30 – 12:00 **A. Dimoulas** *"Rare earth oxides grown by MBE for ultimate scaling"*

12:00 – 13:30 Lunch

**Applications II** (Chair: G. Bersuker)

13:30 – 14:00 **T. T. M. Palstra** "*Magnetoelectric properties of* $REMnO_3$ *compounds*"

14:00 – 14-30 **O. Nilsen** "*Growth and characterization of* $La_xCa_{1-x}MnO_3$"

14:30 – 15:00 **S. Bär** "*Sesquioxides as host materials for rare-earth-based bulk and waveguide lasers*"

**Special discussion**

15:00 – 16:00 "*Outlook on the applications of rare earth oxide thin films"*. Coordinators: M Fanciulli and H. Iwai

16:00 – 16:15 Conclusion and final remarks (**M. Fanciulli**)

## 5. Final list of participants

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## 6. Statistical information on participants

• Total number of participants: 29 people (the two Convenors, a ESF/PESC representative, 21 invitees, and 5 researchers of the MDM-CNR/INFM National Laboratory - the Convenor affiliation).

• Age range of the invetees: two under 30, six between 30 and 40, nine between 40 and 50, two between 50 and 60, and finally two above 60.

• Young investigators among the invetees: four (Lo Nigro, Seguini, Nilsen, and Bär).

• Total number of Women among the invitees: four.

• Countries of origin and nationality of the invitees:
**EC Countries**: Italy, Germany, Finland, United Kingdom, The Netherlands, Norway, Ireland, Greece, and France.
**Non-EC Countries**: Russia, Japan, and United States of America.
All participants were native from their country of origin.

• Number of invitees from Academia and Research institutes: 19
Number of invitees from Industry: 2.

• Sessions: 8.

• Special discussions: 2.

• Interdisciplinarity: Materials Engineering, Physics, Chemistry (in particular Organometallic Chemistry), Device Technology.