



bmb+f - Förderschwerpunkt

Kondensierte Materie

Großgeräte der physikalischen
Grundlagenforschung

Schlussbericht vom 21.07.2004 zum Thema:

Nutzung der Rossendorfer Beschleuniger für ionenstrahlphysikalische Verbundforschungsexperimente

Zuwendungsempfänger:	Forschungszentrum Rossendorf e.V.
Projektleitung:	Prof. Dr. W. Möller
Förderkennzeichen:	05KK1BRA/7
Förderzeitraum:	01.04.2001 – 31.03.2004
Zuwendung:	180.547,00 EUR
E-Mail:	w.moeller@fz-rossendorf.de
Projektträger:	Projektträger DESY-HS

Genutzte Großgeräte:

FZR - AIM

Angaben zum Projekt:

Veröffentlichungen:	13
Konferenzbeiträge:	6
Diplomarbeiten:	5
Dissertationen:	6
Habilitationen:	1
Patente:	0

Das diesem Bericht zugrundeliegende Vorhaben wurde mit Mitteln des Bundesministeriums für Bildung und Forschung gefördert. Die Verantwortung für den Inhalt dieser Veröffentlichung liegt beim Autor.

Schlussbericht-Kurzfassung

Zuwendungsempfänger: ***Forschungszentrum Rossendorf e.V.***

Projektleitung: ***Prof. Dr. Wolfhard Möller***

Nutzung der Rossendorfer Beschleuniger für ionenstrahlphysikalische Verbundforschungsexperimente

Die Förderung im Rahmen dieses Vorhabens hatte das Ziel, Forschergruppen von deutschen Hochschulen und Universitäten sowie außeruniversitären Forschungseinrichtungen, die über keine eigenen Beschleuniger verfügen, die Nutzung der im Ionenstrahllabor des Forschungszentrum Rossendorf betriebenen Teilchenbeschleuniger zu ermöglichen und durch methodisch-instrumentelle Entwicklungen die Basis für aufwändige Experimente zu sichern, die mit den finanziellen und technischen Möglichkeiten der Nutzer nicht realisierbar gewesen wären.

Genutzt wurde die Beschleuniger im Vorhaben fast ausschließlich für die Ionenstrahlanalytik, die jedoch keine einheitliche, geschlossene experimentelle Methode darstellt, sondern für eine ganze Reihe von sehr unterschiedlichen Techniken steht.

An den Rossendorfer Beschleunigern sind zur Zeit 12 Experimentierplätze installiert, an denen praktisch alle Methoden der ionenstrahlanalytischen Festkörpercharakterisierung zur Verfügung stehen. Dazu zählen neben den Standard-Verfahren wie Rutherford – Rück-Streuspektrometrie (RBS) /channeling, teilcheninduzierter Röntgenemissionsspektrometrie (PIXE) und der Kernreaktionsanalytik (NRA) auch Experimentiereinrichtungen, die den Höchststand in der Welt mitbestimmen wie der externe Protonenstrahl, der mit einer Kombination von PIXE, PIGE und RBS i.w. zur Analytik von Kunstgut eingesetzt wird, zwei Magnetspektrometer für die höchstauflösende Teilchenspektrometrie und die in-situ ERDA, die transiente Prozesse nahe der Oberfläche von Festkörpern mit einer Zeitauflösung im Sekundenbereich erfassen kann.

Alle diese Einrichtungen standen für Arbeiten von Gastwissenschaftlern zur Verfügung und wurden unterschiedlich häufig eingesetzt.

Im Berichtszeitraum wurde auch eine neue UHV-Apparatur an der ECR-Quelle intensiv für Untersuchungen zur Wechselwirkung hochgeladener langsamer Ionen mit Festkörperoberflächen genutzt.

Die wissenschaftlichen Themen, die von den Gastgruppen bearbeitet wurden, sind sehr unterschiedlich. Sie reichen von der Element -Analytik von Kunst- und Museumsgut über atomare Transportvorgänge an Grenzflächen und der Struktur von heteroepitaktischen Dünnschichten bis zu Grundlagenuntersuchungen der Ion-Festkörper-Wechselwirkung.

Es ist deshalb nicht möglich, die erzielten Ergebnisse in einem Bericht zusammenzufassen, sondern es wird hier ausdrücklich auf die Literaturliste verwiesen, die Teil des Schlussberichtes ist.



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Schlussbericht

Zuwendungsempfänger: **Forschungszentrum Rossendorf e.V.**

Projektleitung: **Prof. Dr. Wolfhard Möller**

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Die wissenschaftlichen Themen, die von den Gastgruppen bearbeitet wurden, sind sehr unterschiedlich. Sie reichen von Grundlagenuntersuchungen der Ion-Festkörper-Wechselwirkung über Experimente zu atomaren Transportvorgänge an Grenzflächen oder zur Struktur von heteroepitaktischen Dünnschichten bis zu der Element -Analytik von Kunst- und Museumsgut

Es ist deshalb nicht möglich, die erzielten Ergebnisse in einem einheitlichen Bericht zusammenzufassen.

Vielmehr wird hier versucht, an ausgewählten Ergebnissen diese thematische Breite und den Erfolg des Vorhabens zu dokumentieren. Es wird hier ausdrücklich auf die die Publikationen (s. Literaturliste) verwiesen, die Teil des Schlussberichtes ist.

Über den Untertiteln ist jeweils die hauptsächliche Nutzergruppe vermerkt, die Zahl der Personen, die an den Experimenten im Verlauf des Projektes teilgenommen haben, ist sehr groß und deshalb wird keine Namensliste angefügt.

Ausgewählte Ergebnisse

1. Grundlagenuntersuchungen zur Ion-Festkörper-Wechselwirkung

TU Dresden

Charge state distributions of heavy ions after a close collision with surface atoms

Magnetic analysers used in high-resolution RBS separate ions of equal energy but different charge. In the case of near surface scattering the charge state distribution of the emerging ions can deviate significantly from the equilibrium distribution. To convert the energy spectra into depth profiles the knowledge of the charge state distributions (CSD) of the emerging ions as a function of depth is necessary. As a special case, charge state distributions resulting from a single collision of the primary ion with the target atom are of importance in the analysis of very thin surface films. Therefore distributions for Li, C and F ions scattered under single collision conditions at Au atoms were measured at energies below 500 keV/amu. To this end sub-monolayers of Au were

deposited onto a clean Si sample in a UHV scattering chamber. The measured distributions were compared with those resulting from scattering at thick targets, where the charge state distribution is expected to be in equilibrium. It is found that the equilibrium mean charge state for Li-ions is smaller than the mean charge state resulting from a single collision. For C and F ions the opposite behaviour is observed. While the difference is very small for C ions it is pronounced for F ions where the corresponding distributions are shifted by about one charge unit. For Li and C ions the difference between mean equilibrium charge state and the mean charge state after a single collision is almost constant in the energy range considered. Whereas for F ions a slight decrease at higher energies is found. In conclusion the trend of an increasing difference between equilibrium and single collision charge state distributions with increasing nuclear charge presumed earlier could be corroborated.

TU Dresden

Potential energy retention of highly charged argon ions in Au surfaces

For a comparative study for different solid surfaces, the retention of potential energy of multiple charged Ar ions into clean Au surfaces was studied. With a calorimetric setup the target temperature increase was measured during the bombardment with Ar^{q+} ($q=1-8$) ions with kinetic energies varying between 60 eV/q and 200 eV/q. Extrapolating to zero kinetic energy, the deposited potential energy is separated from the deposited kinetic fraction. The potential energy retention coefficient into clean Au surfaces results as 0.9 ± 0.1 , almost independent on the charge state of the ions. Compared to former results obtained on Si (0.6 ± 0.2) and on unprepared Cu surfaces (0.2 ± 0.1) the retention in clean Au surfaces is considerable higher. Additionally, in order to identify the differences in the relaxation mechanisms on the different surfaces, the amount of energy was measured, which is released from Si and Au surfaces during the ion-surface interaction by secondary electrons. First results of the measured integral electron emission show a dependence of the re-emitted energy on the kinetic energy of the multiple charge ions.

2. Instrumentierung

Forschungszentrum Karlsruhe

A compact AMS facility for tritium depth profiling

Depth profiling measurements of tritium in carbon samples have been performed at a dedicated AMS facility equipped in a first step with an air-insulated 100 kV tandem accelerator. The samples were cut from the inner wall of the fusion experiment JET Culham/UK and prepared in the Forschungszentrum Karlsruhe. At the air-insulated 100 kV tandem accelerator both a gas stripper and diamond-like carbon (DLC) stripper foils have been tested. After successful tests of the long-lived DLC stripper foils a compact SF_6 -insulated 100 kV tandem accelerator was constructed and installed in the AMS facility. The 15° analysing magnet was replaced by a 90° magnet to reduce some disturbing background signals. Test measurements at this compact AMS facility have been performed with standard samples from the LLNL Livermore/USA (TiH_2) and from the Tritium Laboratory of the IFIN-Horia Hulubei Bucharest/Romania (tritium in carbon). Depth profiles of other light elements (D, Li, Be, C, ...) can be measured simultaneously in a SIMS mode without acceleration of the considered ions using the 100 kV tandem.

The Rossendorf small magnetic spectrometers for high resolution RBS/ERDA

The increasing importance of ultra-thin layers for novel technologies demands quantitative analysis techniques with a depth resolution of atomic monolayers, which can be obtained for RBS and ERDA by magnetic spectrometers. At the 3 MV Tandetron accelerator a magnetic spectrometer, consisting of an UHV scattering chamber and a simple dipole magnet with circular field boundaries (Browne–Buechner spectrometer). Since in many cases of high-resolution ion beam analysis the samples must be prepared in situ in UHV, the chamber with a base vacuum of $4 \cdot 10^{-10}$ mbar is equipped with an ion sputter gun and two low rate e-beam evaporators for in situ layer deposition. A RHEED system is used to check the surface reconstruction and monitor the layer growth. Samples are transferred, together with a BN heater, to the precision 5-axes channelling goniometer. The magnet with a mean radius of 0.65 m is mounted vertically and can be positioned either at 35.5° or 144.5° . The backward position offers the advantage of a high mass resolution, but the Rutherford cross sections are a factor of about 100 lower than at the forward angle, which is the preferred position if kinematically possible. At the 5 MV tandem accelerator a QQDS magnetic spectrometer is being installed. The facilities for in situ sample preparation in UHV are similar.

3. Dünne Schichten , Epitaxie, atomarer Transport

TU Chemnitz

Surfactant Mediated Growth of Mn_4Si_7 Layers on (001)Si

Semiconducting silicides are candidates for the use in low-scale thermoelectric devices. Among them higher manganese silicides (HMS or $MnSi_{1.7}$) are the most promising thermoelectric silicides of p-type. Furthermore, this material is under discussion for optoelectronic application. However, its complicated tetragonal crystal structure and the existence of a number of similar $MnSi_x$ phases with a composition x ranging from 1.67 to 1.75 [1] have hindered their extensive practical use in thin film technologies. Considering the results obtained in our earlier investigations the shortest in the c-axis length HMS phase (Mn_4Si_7) is used in the following discussion to explain the obtained results.

It has been shown recently that deposition of Mn onto (001)Si at substrate temperatures higher than 600°C leads to the formation of large silicide islands growing on the substrate surface with the major part of their elongated grains parallel to $\langle 110 \rangle_{\text{Si}}$ (Fig. 1(a)) [3]. XRD investigations show the observed silicide islands to exhibit the following texture: $(110)Mn_4Si_7 \parallel (001)Si$.

The found island morphology of the Mn_4Si_7 films can be modified by the deposition of about one monolayer of Sb as a surfactant (surface active substances) onto (001)Si prior to the Mn-deposition. This process results in an increase of the silicide island density by about two orders of magnitude and decrease of the silicide grain dimensions to nanometer range .

Furthermore, applying of Sb leads to a change in the crystalline orientation of the silicide grains. Electron diffraction investigations have shown that in the presence of Sb the silicide layers grow with the preferential orientations: $(100)[010]Mn_4Si_7 \parallel (001)[100]Si$ (as well as $(010)[\bar{1}00]Mn_4Si_7 \parallel (001)[100]Si$. The preferentially oriented silicide grains have been found to grow into the Si matrix with some of their grains parallel to $\langle 100 \rangle_{\text{Si}}$. They are observed to have smooth $(001)Mn_4Si_7/(010)Si$ interfaces as well as Mn_4Si_7/Si interfaces, which are inclined to (001)Si by angles ranging from 15° to 25° (Fig. 4).

RBS investigations have shown the Sb atoms to segregate to the surface of the growing layer. Within the detection limit of RBS it can be concluded that in our experiment the Sb atoms are incorporated neither into the silicide nor into the silicide/Si interface.

Ruhr-Universität Bochum, Experimentalphysik III, Bochum
Microchanneling investigation of β -FeSi₂-structures

The suggested direct band gap of 0.8 eV renders β -FeSi₂ a promising candidate for silicon based μ -LED's. Monocrystalline β -FeSi₂ layers would even open possibilities for μ -laserdiodes. However, the synthesis of these structures appears to be difficult. The combination of the ion projector at the Ruhr University of Bochum and the μ -Channeling set-up at FZR allows a fast synthesis as well as the analysis of such layers. First μ C-RBS analyses of FeSi₂-structures, produced by Fe implantation into Si at an ion energy of 800 keV at different implantation temperatures and with structure sizes of 70-190 μ m, demonstrate that bulk formation by Ostwald ripening depends on implantation temperature and structure size. Even if the layers do not show the desired crystalline characteristics, valuable information could be obtained on the damage and the layer structures. Additional Raman measurements showed clear fingerprints of β -FeSi₂.

Institut für Physik, Technische Universität Chemnitz

Study of compositional, structural and mechanical properties of BCN layers produced by reactive dc magnetron sputtering

Within the triangle B-C-N some materials with exceptional mechanical, electrical and optical properties were found, like e.g. diamond, c-BN, DLC or B₄C. Recently efforts were made to combine all of these elements to produce thin layers with superior properties. Layers of the full ternary system B-C-N with thicknesses in the range of 800 – 2000 nm were deposited on silicon by reactive dc magnetron sputtering. In order to vary the B/C ratio different sputter targets like BC₄, BC and B₄C as well as pure boron and carbon targets were used. To add on nitrogen the sputtering process occurred in a reactive atmosphere consisting of a N₂/Ar mixture with a total pressure of 0.46 Pa. The substrate potential was floating or fixed between 100 and 400 V to study the influence of ion energy on the layer growth. The elemental compositions of the layers were determined by ERDA using 35 MeV Cl⁷⁺ ions. The mechanical properties of the layers were characterised by determination of hardness and Young's modulus and their bonding characteristics by FTIR measurements. For substrate temperatures up to 100 °C the B/C ratio of the sputter target is reproduced in the layer and the N-content increases linearly with the N₂/Ar ratio. At higher temperatures the formation of volatile CN-compounds obviously leads to boron rich layers. By adjusting the composition of the layers their mechanical properties can be varied by a factor of three, whereas the ion bombardment leads to a variation by a factor of two.

Institut für Festkörper- und Werkstofforschung Dresden

Effect of ⁵⁶Fe- and ⁵⁸Ni-ion implantation on the magnetoresistance of La_{0.7}Sr_{0.3}MnO₃ thin films

$\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ is of high interest because of its extremely large negative magnetoresitivity (MR) near the Curie temperature where also a transition from semiconducting to metallic conductivity occurs. Implantation of Fe and Ni with increasing fluence leads to an increase of resistivity and a decrease of the temperature corresponding to the maximum MR. Whereas this maximum occurs at about 360 K for the as-deposited films, the MR at 300 K, as a parameter important for potential applications, can be enhanced by implantation. The transition to insulator- or semiconductor-like behaviour observed at low temperatures for medium and higher fluences, respectively, is strongly indicative for the coexistence of ferromagnetic metallic and antiferromagnetic or spin-canted insulating regions where the current is realized by percolative transport between ferromagnetic metallic clusters.

TU Bergakademie Freiberg, Lagerstättenlehre

Micro PIXE analysis of trace elements in sulfide ore minerals

Trace element distributions in principal ore-forming minerals play an increasingly important role in exploration of mineral deposits. The incorporation of trace elements into mineral assemblage occurs at the time of formation and preserves the physical and chemical properties of the mineralizing fluids. In this study sulfide mineral samples of the porphyry-epithermal environment were investigated to identify mineral hosts and mineralogical sitings of the trace elements Bi, Se, Te, Ag, In and Sn. In polished thin sections of representative sulfide minerals the major element composition of expected mineral host were identified by EDX whereas the trace elements were detected by micro PIXE at the Rossendorf nuclear microprobe. The trace element concentrations were calculated by the Guelph PIXE software package fitting the trace elements in a matrix known from previous electron microprobe studies for pyrite, chalcopyrite, sphalerite, galena, bornite, digenite, enargite and luzonite.

TU Dresden, Institut für Werkstoffwissenschaft; TU Dresden, Institut für Kristallographie und Festkörperphysik
Structural and magnetic phase transformation in metastable Fe-Cr alloys induced by ion irradiation

Unusual metastable paramagnetic phases have been observed in Fe-Cr thin films (~ 40 nm) fabricated by pulsed laser deposition. Structural and magnetic phase transformations in these alloys induced by ion irradiation have been observed. The critical dose for the transformation to the more stable b.c.c. structure depends on the initial phase of the film and the ion mass. The body-centered tetragonal phase which forms at low Cr content (~30 at.%) can be completely transformed to the b.c.c. phase by a dose of 5×10^{15} Cr/cm² whereas the primitive orthorhombic phase of roughly equiatomic Fe-Cr alloys is about 4 times more resistant against ion bombardment. A five times higher Ne-ion dose is required to induce the same transformation as by the Cr bombardment. The formed Fe-rich b.c.c. phase is ferromagnetic. A ferromagnetic pattern, written by a fine-focused Cr-ion beam in a paramagnetic alloy (face-centered orthorhombic, 37 at.% Cr) using an alloy liquid metal ion source ($\text{Er}_{70}\text{Fe}_{22}\text{Ni}_5\text{Cr}_3$), could be detected by magnetic force microscopy.

TU Dresden, Institut für Werkstoffwissenschaft

Influence of the polishing procedure on the hydrogen content of Ti and Ti alloy surfaces

During the mechanical polishing of Ti and Ti alloys a so-called Beilby layer is generated, which may contain an increased concentration of H. Being detrimental for medical implant applications, the presence of H is associated with a degradation of the mechanical properties due to H embrittlement as well as increased bacterial growth. In order to investigate the influence of the polishing procedure on the H content of Beilby layers, the H depth distribution was analysed in mechanically polished samples of c.p.-Ti, Ti6Al7Nb, Ti6Al4V, and Ti15Mo (grinded by wet SiC paper and polished by SiO₂ colloid) from three different laboratories using the ¹H(¹⁵N,

$\alpha\gamma$)¹²C nuclear reaction. Strongly different H concentrations (2 – 60 at.%) in layers from about 5 to 400 nm were observed, being attributed to different procedures of polishing.

4. Externer Protonenstrahl / Kunst- und Museumsgut

TU Bergakademie Freiberg, Archäometrie

The “Sky Disk of Nebra”: Non-destructive gold composition analysis

The spectacular bronze disk of 32 cm diameter and 2 kg weight (dated 1600 B.C.) is supposed to represent the world oldest image of the star sky. Green patina coating, naturally grown, is interrupted by numerous gold applications showing the sun and the moon set in a field of 30 glimmering gold stars, a golden horizontal arc (one lost) as well as another arc interpreted as a barque. Archaeologists are interested whether all the gold applications have one and the same composition. PIXE on air was chosen for non-destructive characterization of the silver and copper containing gold foils. Two PIXE detectors were used for simultaneous measurements: PIXE1 (no filter) for survey analysis and low-Z constituents, PIXE2 (30 μm Zn absorber) with selective filtering of the intense Au-L radiation for improvement of the Ag-K/Au-L intensity ratio. As found, only the barque does not match the otherwise consistent gold composition.

Rheinisches Archiv- und Museumsamt, Abtei Brauweiler, Pulheim,

Inks on parchments – characterisation using external PIXE analysis

Interesting notations accompanied by characteristic initials were found on the rear sides of ancient documents¹. 4 MeV external proton beam PIXE was used for the identification of different mixtures of ferro-gallic ink, in particular from the Fe/Cu and Fe/Zn X-ray intensity ratios. With one exception the inks of the master texts were proven to differ significantly from those of the comments and the initials on the reverses of the parchments. Three documents, characterised by the visually identical initials “TV”, have been of special interest. Here, identical Fe/Cu and Fe/Zn X-ray intensity ratios were obtained from all the “T” and “V” letters. However, Hg as a striking element in the PIXE spectrum was observed only in two cases. As known from the literature, preparation of the inner walls of the inkpots with a mercury sublimate is of advantage in order to prevent the ink from moulding. Supposing mercury to be concentrated in the bottom of the barrel, Hg containing ink of the initials can be interpreted as resulting from deep dunking of the writing utensil into the inkpot. This assumption is corroborated by high absolute Fe and Cu X-ray intensities arising from the viscous ink consistency at the bottom of the inkpot.

Laboratory of scientific art analysis Dresden

Non-destructive surface characterization of a historic church window

Visual inspection of historic windows (St. Marien Church Rostock) gave restorers the impression that the outside glass front might have been covered originally by a light attenuating organic painting material. This assumption was supported by chemical protein analysis showing a slight positive reaction. The hypothetical organic layer was visible on a 5 mm wide glass strip along the edge of the fretted lead. The adjacent large surface region was covered by corrosion products due to natural weathering. Analyses were performed using the combination of RBS and PIXE at the external proton beam ($E_p = 4$ MeV, $I_p \sim 200$ pA). On the bare glass strip RBS identified a thin Pb and Fe containing surface layer. PIXE proved that Si-K-radiation from the glass bulk was attenuated by this top layer. On the weathered glass area RBS-PIXE analysis found an extended surface layer containing Pb

(leaching of the fretted lead) and Fe (pollution from metallic struts). From both results it was straightforward to identify the questionable painting material as a thin layer of initial corrosion. Obviously, the weathering process was suppressed in the narrow glass region due to its protection from the adjacent fretted lead.

Fachhochschule Köln, Fachbereich Restaurierung, gemeinsam mit Staatliche Kunstsammlungen Dresden, Kupferstich-Kabinett,

Identification of tools used for creating historic hand drawings

For the study of drawing materials on paper the nearly background-free X-ray spectrum qualifies PIXE as a well-suited method. Proton beam intensities of only 200 pA and acquisition times of at most 30 seconds are sufficient to get significant signal from the applied substances, using the large area (80 mm²) Si(Li) detector being available at the Rossendorf external beam set-up. Delicate hand drawings of the 15th – 17th century from the "Kupferstich-Kabinett Dresden" were under analysis. The interest of arts scientists relates to pigments of soft contours, light subfonts and gentle washouts. The challenge was to identify the types of these different painting materials on the very sensitive paper backing. Thus, lines from red chalk and silver pens were found. It was of special interest to reveal brownish and greyish tints which could be identified as ferro-gallic ink and lead from lead pens, respectively. In addition, PIXE enables to distinguish graphic tools made from carbon black (no characteristic X-ray line) and mineral black (Al and Si peaks). The simultaneous detection of RBS spectra allows to visualise individual techniques of paper grounding by chalk containing mash.

Schlussbericht - Veröffentlichungen

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R.Grötzschel, C.Klein,M.Mäder

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20th International Conference on Atomic Collisions In Solids
PURI (Orissa), INDIA, 19th-24th Jan 2003

C. Neelmeijer, M. Mäder

Reverse painting on glass as seen by the proton beam

Int. Workshop on the use of ion beams in materials sciences, medicine and archaeometry,
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