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Rosa Córdoba Castillo

Functional Nanostructures Fabricated by Focused Electron/Ion Beam Induced Deposition

Doctoral Thesis accepted by
the University of Zaragoza, Spain

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Book Chapter

29. J. M. De Teresa, A. Fernández-Pacheco, R. Córdoba, M. R. Ibarra, Chapter 23 "Electrical transport properties of metallic nanowires and nanoconstrictions created by FIB", pp. 668–685; in Nanofabrication Using Focused Ion and Electron Beams: Principles and Applications (2012). Editors: P. E. Russell, I. Utke, S. Moshkalev, Oxford University Press. ISBN: 9780199734214.

Supervisors’ Foreword

The use of focused electron and ion beams has certainly changed the way we image and pattern matter at small scales. Due to the high acceleration voltage in the kV range, such focused beams show short wavelengths due to the De Broglie relationship, and thus, are not limited by diffraction effects for practical purposes. This fact has encouraged researchers to build sophisticated instruments based on focused beams such as the Scanning Transmission Electron Microscope, with current imaging resolution below 0.1 nm. For micro- and nano-fabrication, the Scanning Electron Microscope (SEM), currently producing beam spots down to 1 nm size, and the Focused Ion Beam (FIB), currently producing beam spots down to 3 nm size, have become essential tools in Materials Science and Nanoscience laboratories. In particular, existing commercial or home-made equipment integrating FIB-SEM columns plus additional parts such as Gas Injection Systems (GIS) have become popular not only in the Semiconductor Industry for circuit edit and mask repair but also in research laboratories for many different applications like lamellae preparation, cross-sectional imaging, subtractive nanopatterning, establishment of electrical connections to nanostructures, in-situ nanomechanical studies, the growth of functional optical, magnetic, conductive, insulating, semiconductor and superconductor nanostructures, etc. Thus, several one-step lithography processes can be done in a FIB-SEM without the painful use of resist-based processing, opening new routes for nanofabrication.

Rosa Córdoba’s Ph.D. thesis exploits the use of FIB-SEM equipment including GIS for the growth of tailored magnetic and superconducting nanostructures. The used techniques are coined Focused Electron Beam Induced Deposition (FEBID) and Focused Ion Beam Induced Deposition (FIBID). In her thesis, Dr. Córdoba has produced and characterized high-quality ferromagnetic nanostructures grown by FEBID and using $Co_2(CO)_8$ and $Fe_2(CO)_9$ as precursor molecules. By changing growth parameters such as beam current and voltage, flux pressure, dwell time, substrate temperature, etc., she has been able to achieve Co magnetic deposits with high metal content (>95 %) and high resolution (30 nm). Those deposits were subsequently found to be functional from the magnetic point of view, with great potential for their integration in magnetic sensors, memories, and logic. The microstructure of these cobalt deposits was thoroughly characterized by her and the role of autocatalytic effects during certain growth conditions unveiled. On the other hand, the Fe magnetic deposits could not be grown with such purity but showed interesting magnetotransport properties such as a Giant Anomalous Hall Effect. The second part of the thesis focused on the growth and characterization of nanostructured grown by FIBID and using $W(CO)_6$ as precursor molecules. In 2004 it was discovered that the resulting material is a superconductor with a critical temperature around 5 K. The important applications of the FIBID technique in the semiconductor industry were then also applicable for superconducting electronic devices. This thesis includes a complete characterization of this new superconductor, and the influence of the growing conditions. It is shown that the material is amorphous, presenting very low pinning for the superconducting vortices and excellent characteristics for studying the vortex lattice using Scanning Tunneling Microscopy. Then, she explored the limits of the FIBID technique and could fabricate the narrowest, but still continuous, lines of 50 nm. This achievement has permitted the observation of fascinating new size effects in the dynamics of vortices that appeared during the magnetotransport experiments presented in this thesis.

Zaragoza, July 2013

Prof. José M. De Teresa
Prof. Javier Sesé

Contents

Acronyms

AFM	Atomic force microscopy
AHE	Anomalous Hall effect
AMR	Anisotropic magnetoresistance
BCS	Bardeen–Cooper–Schrieffer
BE	Binding energy
BSE	Backscattered electron
CASINO	Monte Carlo simulation of electrons in solids
CCD	Charge-coupled device
CDEM	Continuous-dynode electron multiplier
CNT	Carbon nanotube
CVD	Chemical vapor deposition
DC	Direct current
DOS	Density of states
DT	Dwell time
EBL	Electron beam lithography
EDS	Energy dispersive X-ray spectroscopy
EELS	Electron energy loss spectroscopy
ELNES	Energy-loss near-edge structure
ES-VRH	Efros–Shklovskii variable range hopping
ETD	Everhart–Thornley detector
fcc	Face-centered cubic
FEB	Focused electron beam
FEBID	Focused electron beam-induced deposition
FEBIE	Focused electron beam-induced etching
FEG	Field emission gun
FFT	Fast Fourier transform
FIB	Focused ion beam
FIBID	Focused ion beam-induced deposition
FIBIE	Focused ion beam-induced etching
GAHE	Giant anomalous Hall effect
GIS	Gas injection system
GMR	Giant magnetoresistance
HAADF	High angle annular dark field
hcp	Hexagonal close packed

HDD	Hard disk drive
HE	Hall effect
HRTEM	High-resolution transmission electron microscopy
HV	High vacuum
ITMR	Intergrain tunneling magnetoresistance
LAMH	Langer–Ambegaokar–McCumber–Halperin
LMIS	Liquid metal ion source
MBE	Molecular beam epitaxy
MBJ	Mechanical break junction
MEMS	Microelectromechanical systems
MFM	Magnetic force microscopy
MIT	Metal–insulator transition
MOKE	Magneto-optic Kerr effect
MR	Magnetoresistance
MRAM	Magnetic random access memory
M_s	Saturation magnetization
MTJ	Magnetic tunnel junction
NEMS	Nanoelectromechanical systems
NW	Nanowire
OHE	Ordinary Hall effect
PCA	Principal component analysis
PE	Primary electron
PLD	Pulsed laser deposition
PPMS	Physical properties measurement system
PS	Phase slip
QPSC	Quantum phase slip center
RAM	Random access memory
RGA	Residual gas analyzer
RIE	Reactive ion etching
RRR	Residual resistivity ratio
RT	Refresh time
SC	Superconductor
SE	Secondary electron
SEM	Scanning electron microscopy
SI	Secondary ion
SQB	Superconducting quantum bit
SQUID	Superconducting quantum interference device
SRIM	Stopping and range of ions in matter
SSPD	Superconducting single-photon detector
STEM	Scanning transmission electron microscopy
STM	Scanning tunneling microscopy
STS	Scanning tunneling spectroscopy
TAPS	Thermally activated phase slip
TEM	Transmission electron microscopy
TLD	Through-the-lens detector

TMR	Tunneling magnetoresistance
TRIM	Transport of ions in matter
UHV	Ultrahigh vacuum
VD	Volume per dose
VRH	Variable range hopping