5th Brazilian Materials Research Society Meeting Addresses Academia and Industry Interests

http://www.sbpmat.org.br/5meeting/

The 5th Brazilian Materials Research Society Meeting (SBPMat), chaired by Aloisio Nelmo Klein (Universidade Federal de Santa Catarina, Florianópolis), was held in Florianópolis, Brazil, October 8–12, 2006. The technical sessions included 13 symposia covering a range of materials research topics, including surface engineering, powder technology, high-temperature materials, photodevices, hybrid materials, electron microscopy, synchrotron radiation, electroceramics, nanostructured biological materials, forensic science, microscopy for industry, advances in microscopy for materials characterization, and computational techniques for materials design. Plenary speakers delivered overviews of specific materials research areas and opportunities for international collaborations. The meeting also featured poster sessions and a roundtable discussion on "Industrial Development versus Academic Research in Brazil: New Trends."

The meeting opened with a lecture by **Peter F. Green** (Univ. of Michigan), the 2006 president of the Materials Research Society. In his lecture, "The Last 100 Years in Materials Research and New Challenges," Green showed how the field of materials science and engineering (MS&E), with its roots in metallurgy, has evolved during the last 50 years, initially due to the influence of physics and chemistry, and now increasingly with biology. Among the exciting possibilities for the future, Green said, is the development of materials by design based on specific functionalities. Green described several grand challenges in the field, including the interface between medicine and materials, biomolecular materials, Moore's law, and nanotechnology. With these developments in the field come changes in materials research education. Green said in many universities around the world, significant materials research is conducted not only in MS&E departments but also in other areas of engineering as well as in the natural sciences and medicine. Similar trends exist in corporate laboratories. Green said that the community has to be aware of and tackle the associated issues and challenges to ensure the continued health of the field of MS&E.

Plenary Lectures Address Quantum Computing, Bioceramics, Instrumentation, and International Collaborations

The development of quantum computers is generating much interest among researchers in fields such as quantum physics, computer science, engineering, and materials science. **Luis Davidovich** (Univ. Federal do Rio de Janeiro, Brazil), in his plenary address, explained two major motivations for this interest: the tremendous recent progress in quantum manipulation of atoms, and the limits on classical computational techniques. Materials research aspects to this field involve what Davidovich called "entangled states," including photonic entangled states. Davidovich gave an overview of papers that have been published on atom–photon entanglement, entanglement of trapped ions, and the entangled quantum state of magnetic dipoles. The issue of entanglement is crucial for quantum information exchange and quantum computing. There are several candidate experimental technologies for quantum computing, including ion traps, quantum dots, nuclear magnetic resonance, optical lattices, and Josephson junctions. The problem with all of these is decoherence. Davidovich suggested that the new experimental techniques are promising for quantum computing. He said that some aspects of quantum cryptography and quantum computers have already been demonstrated.

Plenary speaker **Georg Grathwohl** (Univ. of Bremen, Germany) talked about ceramics for biological applications. Ceramics have been developed for high-performance applications over the last two or three decades. These efforts were instrumental when bioceramics started to be designed for special functions in biomedical applications. There is, however, a much broader approach if inorganic nonmetallic structures are considered for interactions with living organisms. Bioceramics may be defined according to various principles when technical ceramics come into contact with biological environments. Four groups of bioceramics can be identified following different functions of both components, that is, the inorganic and bioorganic parts. These functions are related to biogenesis of hard materials, transfer of biological structures to form ceramics (biomorphology), synergistic interactions in biocers (inorganic/bioorganic hybrids and composites), and bio-related functions of ceramics incorporated into biological systems. Biocers make up a new class of biologically modified ceramics, and the properties of biocers open new technical pathways in biotechnology, process engineering, and related fields. The conversion of biological structures into ceramic parts follows two paths; there is interest in preserving the original biological structure and morphology, such as observed in fibers, wood, and other biological precursors, in the new ceramic materials. However, this also allows for the fabrication of large-scale components such as panels, tubes, and substrates that cannot be produced by more conventional ceramic technologies. Grathwohl concluded by stressing that the target applications of bioceramics interconnect the four different classes, and these materials have significant potential in the future.

With high accuracy, electron energy loss spectroscopy (EELS) can be used to map the nature, position, and bonding of atoms responsible for structural properties of materials, including defects, interfaces, and nanomaterials. In his plenary address, **Christian Colliex** (Univ. Paris-Sud, France) explained the details of EELS measurements using scanning transmission electron microscopy (STEM). He focused on nanoscale materials and described how the EELS technique is ideal for obtaining topographic, atomic-structural, and chemical information. He described the use of core-loss EELS spectroscopy for elemental mapping, bond mapping, and valence-state determination, and showed various results, for example, for BN nanotubes and nanoparticles. He described how EELS can be used to determine interface states using the energy loss near-edge fine structure. Colliex said that future work in the field involves improving the spatial resolution and the energy resolution at very low energy losses.

Two plenary speakers described opportunities for international collaborations in materials research. The Inter-American Materials Collaboration (CIAM) partnership—a program within the U.S. National Science Foundation (NSF)—was described by **Thomas Weber**, director of the Office of International Science and Engineering at NSF. Started in 2003, CIAM involves the cooperation of nine countries in the Americas. The program particularly stresses education, including international exchanges of graduate students and young scientists. Weber also detailed the Pan-American Advanced Studies Institutes (PASI) in collaboration with Latin American countries for short courses for

https://doi.org/10.1557/mrs2007.16 Published online by Cambridge University Press

young scientists, and he described the International Research Fellowship Program for U.S. citizens and residents for postdoctoral work outside the United States. More information is available at the NSF Web site, www.nsf.gov, or by contacting Weber at tweber@nsf.gov.

Anthony Cheetham described the International Center for Materials Research (ICMR), which he co-founded at the University of California, Santa Barbara (UCSB). The stated mission of the ICMR is to promote global excellence in materials science and engineering through research and educational programs. In addition to international materials research collaborations, the center aims to provide international experience for young scientists as well as enhance the capacity of developing countries in materials science by engagement and network building. The international activities of ICMR include collaborative institutions, exchange programs for students from both U.S. and non-U.S. institutions, workshops, and summer schools conducted at UCSB and elsewhere. More information can be accessed from the ICMR Web site at www.icmr.ucsb.edu.

For further details on the research reported at the 5th Brazilian Materials Research Society Meeting, see the symposium summaries that follow. Meeting highlights are also available online at www.mrs.org, under Materials Connections.

Tribology and Surface Engineering

D. Rigney (Ohio State Univ., USA), a keynote speaker for Symposium A on Tribology and Surface Engineering, gave a presentation on sliding friction and wear that included molecular dynamics simulations of effects that occur when two materials slide past one another. Rigney showed movies in which clear vortices formed at the interface between two materials. He compared these with similar patterns in fluid flow, as sometimes revealed by clouds in the atmosphere. The addition of soft, lubricating particles to one of the materials improved the lubrication properties even after mixing. High-energy hard particles tend to clump at the interface and could clearly be seen to rotate as clumps. Future work includes the validation of molecular dynamics predictions via high-velocity sliding and characterization and the development of analytical flow models. The models may be applicable to other situations such as explosive welding, plate tectonics, and realistic wear models, Rigney said.

W. Santana da Silva (Univ. de São Paulo, Brazil) talked about NICRALC, which is a family of casting alloys proposed as a substitute for abrasion- and erosion-resistant cobalt alloys. NICRALC microstructures contain hard chromium carbides dispersed in a continuous Ni-Al-Cr–ordered intermetallic matrix. The motivation for the work was to determine whether NICRALC could substitute for STELLITE 6, a more expensive cobalt-based alloy traditionally used for the same purpose. Mechanical properties of the investment cast alloys were evaluated by compression, hardness, and fracture toughness measurements, all at room temperature. Tribological properties were evaluated by cavitation, rubber-wheel wear, reciprocating wear, and block-on-ring lubricated wear tests. The wear and fracture mechanisms were studied using scanning electron microscopy (SEM) imaging of the worn or cracked surfaces. Results showed that NICRALC is softer than STELLITE and therefore is not as good with regard to tribological behavior. The work is continuing, using SEM and x-ray diffraction studies of the collected debris.

M.R. Cordosa (Inst. de Física de São Carlos, Univ. de São Paulo, Brazil) described the use of a simple laser microstructuring process to replicate the topology of lotus leaves to improve the hydrophobicity of an azopolymer. The azopolymer was exposed to a laser beam interference pattern using a polarized argon-ion laser (488 nm) at 350 mW/cm^2 for 20 min, resulting in a surface relief grating mechanism that caused microstructuring of the polymer. The contact angle of the polymer increased from 85.5° to 90.7°, corresponding to a 5% increase.

Materials for High-Temperature Applications

T.G. Langdon (Univ. of Southern California, USA) presented the opening lecture of Symposium C, Materials for High-Temperature Applications, in which he provided an overview of the processing of ultrafine-grained materials through the application of severe plastic deformation and the corresponding microstructure evolution during processing.

A. Möslang (Karlsruhe Research Center, Germany) presented an overview on the candidate materials for the construction of critical parts in the ITER fusion nuclear reactor facility to be started in France in 2007. Refractory alloys (W-based) and 9%-Cr martensitic-ferritic oxide-dispersion-strengthened (ODS) steels display the necessary characteristics to operate at high levels of radiation in safe conditions and long-term cycles. These steels, in particular, have low potential for radioactive activation and are strengthened by a very fine dispersion of particles with sizes below 5 nm. E. Materna-Morris (Karlsruhe Research Center, Germany) showed microstructural changes that occur in low-activation 8–10% Cr ferritic steels after neutron irradiation—in particular, the development of He bubbles along dislocations revealed by TEM inspection of irradiated samples.

Recrystallization in oxide dispersion-strengthened alloys is an important topic for high-temperature applications. The efficiency of these materials in service depends on how resistant they are to recrystallization. Addressing this gap, H.R.Z. Sandim (Univ. de São Paulo, Brazil) presented results on the recrystallization behavior of the nickel-based ODS superalloy PM-1000. The material displayed partial recrystallization even when annealing was performed at high homologous temperatures. This behavior is related to pinning effects caused by fine particles with sizes below 20 nm dispersed in the matrix. Boundaries located at the prior grain boundaries developed nuclei that grow only to a certain extent. Concurrent recovery is responsible for diminishing the driving force for recrystallization. In related work, A.F. Padilha (Univ. de São Paulo, Brazil) and his group described the 475°C embrittlement of DIN1.4575 and MA-956 ODS ferritic alloys caused by the precipitation or spinodal decomposition of the alpha prime phase, resulting in loss of ductility, toughness, and corrosion resistance of these ferritic steels.

L. Ramanathan (Inst. de Pesquisas Energéticas e Nucleares, Brazil) explained the role of rare-earth oxide coatings on the oxidation resistance of chromia-forming alloys. This topic is relevant, since most of the materials for applications in oxidizing environments contain chromium as an alloying element. R.L. Plaut (Univ. de São Paulo, Brazil) outlined the high-temperature processing and microstructure of wrought austenitic steels. C.A. Nunes (Univ. de São Paulo, Brazil) gave an overview on the application of refractory metals and alloys with an emphasis on the role played by Brazil in terms of research and development in this field and as an important supplier of this class of materials.

Materials for Photodevices

P.L. Souza (Pontifícia Univ. Católica do Rio de Janeiro, Brazil), in Symposium D on Materials for Photodevices, described a novel quantum dot structure for infrared photodetectors. Infrared photodetectors based on intraband transitions in quantum dots (QDIPs) should, in principle,

https://doi.org/10.1557/mrs2007.16 Published online by Cambridge University Press

The 5th Brazilian Materials Research Society Meeting (SBPMat) was held in Florianópolis, Brazil, October 8–12, 2006.

outperform the ones based on quantum wells (QWIPs), Souza said, because of the possibility of absorbing normal incident light and operating at higher temperatures due to the longer electron lifetimes. InAs/InGaAlAs/InGaAs/InP quantum dot infrared photodetectors were grown by metalorganic vapor-phase epitaxy and evaluated. The results demonstrate great potential for operation under normal incidence and temperatures above that of liquid nitrogen.

V.I. Klimov (Los Alamos Natl. Lab, USA) discussed the use of nanodots to obtain two or more excitons (electron–hole pairs) with a single photon of light, thereby producing efficiencies that exceed 100%. Klimov said that the efficiencies can reach 700% in the case of lead selenide nanocrystals. Normally, only one exciton is produced per photon; however, carrier multiplication in the nanocrystals allows for more excitons to be produced per photon, Klimov said.

International Symposium on Hybrid Materials and Their Applications

Since encapsulation of biomolecules in a silica matrix was first demonstrated a decade ago, the field has advanced, with various hormones, proteins, liposomes, and bacteria being encapsulated in porous silica matrices. In Symposium E, the International Symposium on Hybrid Materials and Their Applications, B. Dunn (Univ. of California, Los Angeles, USA), described the use of the sol-gel technique to encapsulate various biomolecules, which retain their characteristic reactivity and chemical function. It has been found that encapsulation within a sol-gel matrix stabilizes the biomolecules, with corresponding improvements in thermal resistance, storage stability, and resistance to chemical environments. Among the numerous potential applications of these materials is the ability to fabricate designer sensors. Dunn described the use of sol-gel–immobilized antibodies to detect cortisol levels in astronauts to monitor their stress levels, which could be critical during space flights, since there currently are no in-flight biosensors available. Dunn also discussed the use of biohybrid materials for forming biofuel cells based on enzyme encapsulation that continuously change the chemical energy of a fuel and oxidant directly to electrical energy by utilizing enzymes to catalyze oxidation and reduction reactions. He described the development of nanostructured electrodes for biofuel cells by integrating a three-dimensional (3D) network of carbon nanotubes acting as 3D nanowiring in the non-conductive silica matrix. The biofuel cells can be integrated to form a battery by connecting the biofuel cells in series and using fluidic chambers. Dunn also described recent work on the development of a photo-biofuel cell.

Other highlights included presentations by A.J. Jackobson (Univ. of Houston, USA) on "Chirality and Flexibility in Hybrid Inorganic–Organic Structures"; A.K. Cheetham (Univ. of California, Santa Barbara, USA), "Recent Developments in Hybrid Inorganic–Organic Framework Materials"; J. Dupont (UFRGS, Brazil), "The Template-Like Effect of Ionic Liquids for the Preparation of Nanoparticles"; P.F. Green (Univ. of Michigan, USA), "The Ordering Transition and Structural Instabilities in Block Copolymer/Nanoparticle"; C.P. de Mello (Univ. Federal de Pernambuco, Brazil), "Use of DNA as Template for Luminescent Polyanil"; and F. Galembeck (Univ. Estadual de Campinas, Brazil), "Adhesion in Heterogeneous Systems: Formation of Nanohybrid Aggregates."

Nanostructures and Defect Analysis by Electron Microscopy

With the current burgeoning interest in nanomaterials and the associated development of a wide range of nanomaterials, it is becoming crucial to be able to determine compositions at the nanoscale, said J. Bentley (Oak Ridge Natl. Lab, USA), as he described the use of analytical electron microscopy techniques to determine nanoscale compositions in symposium F. In particular, Bentley focused on two techniques, energy-filtered TEM and spectrum imaging in STEM mode using energy dispersive x-ray spectroscopy (EDS) and EELS. As examples, Bentley described a systematic study of CoCr(TaPt) magnetic recording media, as well as CoCrPtB alloys and CoCrPt perpendicular media. He described the extraction of elemental maps for $CoPtTiO_2$ perpendicular recording media from the EDS/EELS spectrum image. Significant levels of Co, but no Pt, was observed at the grain boundaries. The TiO_2 stoichiometry could also be determined using quantitative EELS. Bentley also discussed work on reaction ball-milled Y-Ni-O nanostructures for the development of ODS steels. He described the compositional mapping of nanoscale $MgAl_2O_4$ spinel. Bentley said that the future direction is toward deep sub-0.1-nm imaging and EELS with single-atom sensitivity.

Time-resolved high-resolution transmission electron microscopy (HRTEM) results and molecular dynamics simulations of atomistic aspects associated with stretching of Au-Ag nanowires were reported by D. Ugarte (Inst. de Física Gleb Wataghin–Univ. Estadual de Campinas, Brazil; Laboratório Nacional de Luz Síncrotron, Brazil). While atomic-sized pure metal nanowires have been previously obtained by mechanical stretching, limited data are available for alloy nanowires. Ugarte's group used *in situ* HRTEM to focus the electron beam on Au-Ag thin films, with nanobridges forming between the holes. These were then stretched to obtain atomic-scale nanowires. The results revealed that, unlike pure metals, structural defects, primarily twins and stacking faults, are sometimes present at the apexes and very close to the most narrow wire constriction or even within the nanowires. Also, similar to pure nanowires, linear suspended chains of atoms were observed. It was also observed that the atomic-sized Au-Ag alloy nanowires exhibited a spontaneous gold enrichment at the nanojunction region during the wire-thinning process, resulting in a dominant gold-like behavior, even for alloys with minor gold content. This suggests the ability to control the stability or length of the atomic chains by using an appropriate and suitable alloy composition, Ugarte said.

Other highlights included presentations by C.B. Carter (Univ. of Minnesota, USA) on "Use of *In Situ* TEM to Study Nanomaterials"; M.G. Norton (Washington State Univ., USA), "Fabrication and Characterization of Nanostructures for Advanced Energy Applications"; D. Ugarte (Inst. de Física Gleb Wataghin–Univ. Estadual de Campinas, Brazil; Laboratório Nacional de Luz Síncrotron, Brazil), "Nanowires and Suspended Atomic Chains from Au-Ag Alloys"; M. Aindow (Univ. of Connecticut, USA), "Transmission Electron Microscopy Studies of Mesoporous Carbon Aerogels and Carbon/Metal Nanocomposites Produced Using Supercritical Carbon Dioxide Based Process"; P.A. Midgley (Univ. of Cambridge, UK), "Electron Tomography of Nanostructures"; C. Colliex (Univ. Paris-Sud, France), "Sub-nm EELS Mapping of Physical Properties in Individual Nanostructures"; J. Liu (Monsanto Co.), "Understanding the Nature of Nanostructured Heterogeneous Catalysts"; N. Browning (Univ. of California, USA), "Atomic-Scale Transmission Electron Microscopy"; and W.J. Botta Filho (Univ. Federal de São Carlos, Brazil), "Microstructural Characterization of Cu-Based Bulk Metallic Glass Containing Nanoparticles."

5th Brazilian Electroceramics Symposium

R. Muccillo (Inst. de Pesquisas Energeticas e Nucleares), in Symposium H: 5th

Brazilian Electroceramics Symposium, addressed the production of stabilized zirconia compounds that are used for solid-oxide fuel cells. He described how the polyacrylamide method produced powders without secondary phases and, with the use of laser scattering, how the amount of organic compound content depends on the degree of agglomeration.

A varistor is a device used to protect electrical circuits against excessive transient voltages. Varistors typically have non-ohmic *I–V* characteristics. In his presentation, M.A. Ramírez (Univ. Estadual Paulista, Brazil) described continuing work on the fabrication and development of varistor ceramics based on SnO_2. The SnO_2-based system has been shown to have excellent electrical properties, including a high value of the nonlinear coefficient, a high electrical breakdown field (3900 V/cm), and low leakage current (below 90 μA). In this presentation, Ramírez reported properties of varistor samples of the SCNCr system (98.9%SnO_2 + 1%CoO + 0.05%Nb_2O_5 + 0.05Cr_2O_3 [mol%]) with different area-volume (A/V) ratios sintered at 1300°C in an ambient atmosphere. It was shown that the A/V ratio had little effect on the electrical and microstructural properties of the SCNCr system when sintered at 1300°C, thus indicating a very stable system, compared to traditional ZnO-based varistors.

Multiferroics are materials in which at least two of the ferroelectric, ferro/antiferromagnetic, and ferroelastic phases coexist. M. Villegas (CSIC, Madrid, Spain) described a coprecipitation method to form zinc, nickel, and cobalt ferrites, MFe_2O_4 (the ferromagnetic phase), by coprecipitation using cobalt and iron chlorides, using nickel and zinc nitrates as precursors and NaOH as the precipitant agent. Commercial $BaTiO_3$ was used as the ferroelectric phase. The two were mixed to obtain a multiferroic material and these mixtures were sintered at 1200ºC to obtain magnetoelectric materials. A clear magnetoelectric response was obtained. The co-existence of the ferroelectric and ferromagnetic phases was corroborated by x-ray diffraction.

In the area of fuel cells, A. Sin (Pirelli, Italy) described the development of new electrodes from the synthesis of nanoparticles and nanocomposites for cathodes (LaSrCoFeO) and anodes (NiCu+CeGdO). Colloidal and sol-gel chemistry were used to synthesize the raw nanomaterials and the "gentle chemistry" allows the crystallization temperature of oxides to be reduced significantly, thereby obtaining extremely fine materials. It also allowed the deposition of layers of controlled thickness and composition and could be used to commercially produce fuel cells. The cells worked for more than 10 days and the performance improved with time. The optimum anode material was Ni(0.2)Cu(0.8).

E.N.S. Mucillo (Inst. de Pesquisas Energeticas e Nucleares) described the requirements of ceramics for solid-oxide fuel cells: high electronic conductivity, low electronic transference numbers, phase stability from room temperature up to 1100°C, thermal expansion and chemistry compatible with other materials, and good mechanical strength. Mucillo described details of the fabrication process and measurements on the CeGdO material. Her conclusions were that nano-sized powders of Gd-doped ceria can be prepared by the oxalate coprecipitation route and that grain growth proceeds by impurity drag (boundary control) or lattice diffusion (pore control). Electrical resistivity of sintered ceramics is not dependent on most of the synthesis parameters.

Nanostructured Biological Materials

In Symposium I on Nanostructured Biological Materials, H.L. Gomes (Univ. of the Algarve, Portugal) described the development of a technique that uses changes in capacitance to monitor the interaction between immune system cells (hemocytes) and intruder cells (parasites). The work was started and funded because oysters on the Atlantic coast (*Ruditapes decussatus*) were being killed by a parasite, *Perkinsus atlanticus*. It was found that some of the oysters were immune, suggesting differences in their immune system. The parasites show a peculiar behavior: they aggregate in clusters when they feel threatened by the hemocytes. The level of this aggregation may provide a measure of immune system efficiencies in oysters. The cell aggregation causes impedance (capacitance) between a microelectrode and a large reference electrode, which can be monitored.

E. Traversa (Univ. of Rome, Italy) described work on the interactions between materials and biological systems for use in medical and clinical applications. A particular example he investigated was the use of copper nanoparticles to suppress the growth of fungi, which can be harmful pathogens for humans. Polymer-matrix nanocomposites with Cu nanoparticles can be used to form packaging that releases copper at a controlled rate that is harmless to humans but high enough to kill fungi.

2nd Brazilian Symposium on Microscopy Applied to Forensic Science

In forensics, the analysis of firearms and bullets is a critical part of investigations. In Symposium J: 2nd Brazilian Symposium on Microscopy Applied to Forensic Science, A.P.C. Campos (Departamento de Polícia Técnico-Científica, Brazil) outlined the techniques involved in comparing firearms and determining whether bullets and cartridge cases can be linked to a specific firearm. The reproducibility and uniqueness of machining marks on bullets and cartridge cases, also called ballistic fingerprints, allow a firearm examiner to identify a specific weapon used in a crime. The primary instrument used is a comparison micro-

Roundtable Discussion Encourages Academia–Industry Cooperation in Brazil

While scientific research in Brazil is very strong, indications are that not much of it gets into real industrial applications. For example, roughly 1.8% of peer-reviewed scientific publications in the world are by Brazilian researchers, whereas only 0.02% of patents worldwide are from Brazil. This indicates a large gap in the transfer of technology from academia to industry and a large disconnect between the two communities. During a roundtable event on "Industrial Development versus Academic Research in Brazil: New Trends," discussions centered on what could be done to bridge this gap and enable academia and industry to work together. While there were no clear solutions to the problem, there was a consensus that academic researchers and industry in Brazil should work more closely to further develop practical technologies. While 80% of research in Brazil is conducted in universities, the two companies represented in the panel—Petrobras and Embraco—also conduct research and, in addition, fund research in universities. The hope was expressed that cooperation between academic researchers and industry will grow, which would in turn benefit the materials research community in Brazil as well as technological developments as a whole in the country.

The panelists were Angelo Fernando Padilha (Escola Politecnica da USP) and Carlos Alberto Schneider (UFSC) from the academic side, and Antonio Claudio C.M. Sant'Anna (Petrobras) and Roberto Binder (Embraco) from the industrial side.

https://doi.org/10.1557/mrs2007.16 Published online by Cambridge University Press

scope, which allows side-by-side observations of microscopic characteristics. For inconclusive cases, electron microscopy is also used. In recent years, automated comparison systems have become a critical tool in forensic laboratories. Several countries have built databases of ballistic images of bullets and cartridge cases. These systems significantly speed up the comparison process; however, the final decision is still the examiner's.

H. Katterwe (Forensic Science Inst., Germany) discussed the science of restoring serial numbers to metal weapons and to plastic stolen goods. Automatic hardness measurement machines make it possible to detect the regions of increased hardness caused by the stamping of a serial number on the surface of a weapon, even when the number has been filed off. Plastics can be examined by exposing the object to solvents that make the polymer chains swell and the compressed regions under the number expand more than the surrounding regions. In other words, this is a form of shape memory. An interesting discovery occurred when a forensic scientist in Germany was making *lebkuchen* and found that the cloves used in the cookie recipe exposed markings on plastic containers. Clove powder has now become an important tool in forensics.

Laser engraving of serial numbers on firearms is currently the norm for new weapons used by the police in Brazil. Stolen firearms often have their serial numbers obliterated. L. da Silva (Escola Naval–Marinha do Brasil, Brazil) described a procedure for recovering serial numbers from obliterated laser-engraved frames that are usually made of an aluminum alloy. After initial observations, the firearm is cleaned and then examined under a stereomicroscope. Slow, careful, and soft polishing is then used, and the surface is scanned periodically. Da Silva presented a number of examples in which the numbers could be recovered using this method.

Advanced Techniques of Microscopy for Characterization and Modification of Materials–MICROMAT

In Symposium M on Advanced Techniques of Microscopy for Characterization and Modification of Materials–MICROMAT, P.S. Dorozhkin (NT-MDT Co., Russia) described a setup that combines atomic force microscopy (AFM) and confocal Raman microscopy in one instrument. A wide range of scanning probe techniques is supported, including scanning Kelvin microscopy, electrostatic force microscopy, atomic force acoustic microscopy, and several others based on different types of interactions between an AFM probe and the surface. These AFM-based techniques provide extensive information about surface physical properties. On the other hand, confocal Raman microscopy gives information about surface chemical composition.

Combined AFM topography and confocal Raman maps of the same sample area allow the separation of single-walled nanotubes from multiwalled nanotubes and distinguish nanotube material from amorphous carbon residuals. AFM phase imaging together with Raman maps allows separation of different phases in polymer blends; confocal Raman mapping in the z-direction (sample depth) provides information on the thickness of polymer coatings. Full 3D imaging with data analysis is also supported. Other applications include investigating single nanowires acting as optical fibers, mapping stress in silicon structures, and AFM observation of protein structures in air and liquid. The ability to obtain multiple images enables rich investigations of samples, said Dorozhkin. Ultimately, the optical diffraction limit of confocal Raman resolution can be overcome with tip-enhanced Raman scattering. Raman maps with <70-nm resolution were demonstrated.

A.B. da Silva (Max-Planck-Institut für Eisenforschung, Germany) described a new scanning electron microscope–secondary electron backscatter diffraction (SEM–EBSD) setup with a focused ion-beam (FIB) system in a dual-beam microscope. Samples can be investigated by removing successive thin slices of material (minimum thickness, ~50 nm) from the sample surface by sputtering with a high-energy ion beam (Ga^+ source) and examining the surface using EBSD patterns. After the milling process, the sample is tilted to the EBSD position (70°) in order to observe the diffraction pattern. The microstructure of the surface can then be analyzed by conventional 2D EBSD-based orientation microscopy. By successive sputtering and EBSD measurements, the 3D microstructure can be built. The machine can be made to function automatically. Electrodeposited cobalt nickel was investigated to understand the growth of columnar grains. The results shown in the presentation took about a week to obtain. Da Silva said that some of the difficulties with the technique are that the etch rate depends on the grain orientation, so that in some cases a long milling time is required to obtain a smooth surface. The gallium beam tends to interact with some samples, causing, for example, amorphization of a certain phase in the material.

D. Ugarte and V. Oiko (Inst. de Física Gleb Wataghin–Univ. Estadual de Campinas, Brazil; Laboratório Nacional de Luz Síncrotron, Brazil) described the development and application of an inexpensive two-probe tip nanomanipulator for *in situ* experiments in an SEM. The manipulator includes two kinds of movements: coarse, with millimeter range and submicron precision; and fine, with micron range and nanometer precision. Coarse movements are based on parallel guiding spring mechanics, which include no moving parts, so they do not require lubrication. Fine movements are based on piezoelectric elements and give a precise three-axis movement of both probe tips, which have independent electrical supplies, allowing for a wide range of nanomanipulation and nanocharacterization experiments. The system has been used for different kinds of *in situ* experiments such as fabricating high-aspect-ratio AFM tips based on multiwalled carbon nanotubes; collecting, moving, and positioning semiconductor nanowires (50–200 nm in diameter, microns in length) on predefined electrical contacts or special sample sites; and measuring electrical properties of nanowires inside the microscope. Interestingly, only carbon nanotubes are strong enough to provide probes at this scale; diamond cannot be used.

Computational Simulation as a Technique to Design New Materials

Based on theoretical work, E.S. Leite (Cidade Universitária, Brazil) proposed a new class of porous materials called isoreticular metalorganic frameworks (IRMOFs), which consist of an isoreticular framework with a cubic lattice with tetrahedral Zn_4O clusters at the cube vertices and organic dicarboxylate aromatic linkers between the vertices. Each Zn_4O is connected to six linkers with octahedral symmetry. Leite described the work in Symposium N on Computational Simulation as a Technique to Design New Materials. IRMOFs are very versatile materials, since the organic linkers can be easily modified or functionalized. Leite simulated storage using grand canonical Monte Carlo simulations to calculate the adsorption isotherms of the methane within IRMOF-6. Leite said that calculations of this kind allow the investigation of material structures for natural gas and hydrogen storage without the cost of experiments.