ASTM Committee E42 Surface Analysis

Donald Baer
don.baer@pnl.gov

(www.astm.org)

ASTM Committee E42 on Surface Analysis

30 years old

Purpose –more reliable analysis

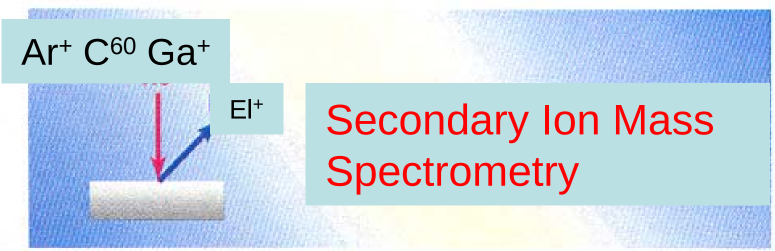
- *Develop Standards*
- *Prepare standard practices*
- *Definitions*
- *Cooperate with other organizations*
- *Advance field – information exchange*
- *Appraise developing methods*

The ASTM Committee E-42 on surface analysis was established in 1976 to guide users of surface analytical instruments in making more reliable analyses. As given on the www.astm.org website, the scope of Committee E-42 on surface analysis is: (a) to review and coordinate the development of standards for all methods of surface analysis by photon, electron, and ion emission or reflection methods which include: AES, XPS, ISS, SIMS, RBS, and the use of ion bombardment and other methods to obtain composition versus depth information, e.g., sputter depth profiling (SDP); (b) to prepare standard practices for the methods included in (a); (c) to establish standard definitions of terms relating to methods included in (a); (d) to cooperate with agencies concerned with the preparation and distribution of standard reference materials; (e) to advance the field of surface analysis by promoting the collection and exchange of information through surveys, cooperative programs, publication of suggested methods and practices, meetings, and symposia; (f) to formulate and evaluate only those standard surface analysis methods that are not included in the scope of other ASTM committees, except by mutual agreement; (g) to maintain a current appraisal of developing surface analysis techniques, as to their principles, applicability, and limitations; and (h) to coordinate the activities in (a)–(g) with other ASTM committees and other organizations having mutual interests.

Charles Evans and Associates (services and literature – bubblechart)
http://www.eaglabs.com/en-US/services/services.html

Lateral Resolution ≈ 10 nm
Information Depth ≈ 10 nm
Depth Resolution ≈ 1 nm

Lateral Resolution ≈ 1 nm
Information Depth ≈ 10 nm



Lateral Resolution (inorganic) ≈ 50 nm
Lateral Resolution (organic) **>** 200 nm
Information Depth ≈ 1 nm
Depth Resolution (inorganic) ≈ 1 nm
Depth Resolution (organic) ≈ 10 nm

Lateral Resolution ≈ 2 μm
Information Depth ≈ 10 nm
Depth Resolution ≈ 1 nm

for
ies

Committee E-42 on Surface Analysis

Subcommittees

E42.01 Editorial

E42.02 Terminology

E42.03 Auger Electron and X-Ray Photoelectron Spectroscopy

E42.06 **Secondary Ion Mass Spectrometry**

E42.08 Ion Beam Sputtering

E42.09 Standard Reference Mat.

E42.11 Standard Reference Data

E42.13 Vacuum Technology

E42.14 Scanning Probe Microscopy (STM/AFM)

US TAG for ISO TC201

Examples
Existing E42 Generated Guides and Standards
AES and XPS

- E827-02 Standard Practice for Indentifying Elements by the Peaks in Auger Electron Spectroscopy
- E902-05 Standard Practice for Checking the Operating Characteristics of X-Ray Photoelectron Spectrometers
- E983-05 Standard Guide for Minimizing Unwanted Electron Beam Effects in Auger Electron Spectroscopy
- E1078-02 Standard Guide for Specimen Preparation and Mounting in Surface Analysis
- E995-04 Standard Guide for Background Subtraction Techniques in Auger Electron Spectroscopy and X-ray Photoelectron Spectroscopy

Committee E42.14 on STM/AFM

E1813-96(2007) Standard Practice for Measuring and Reporting Probe Tip Shape in Scanning Probe Microscopy

E2382-04 Guide to Scanner and Tip Related Artifacts in Scanning Tunneling Microscopy and Atomic Force Microscopy

E2530-06 Standard Practice for Calibrating the Z-Magnification of an Atomic Force Microscope at Subnanometer Displacement Levels Using Si(111) Monatomic Steps

Priority needs established by Community Survey

Thin (nano) layer analysis a very high priority

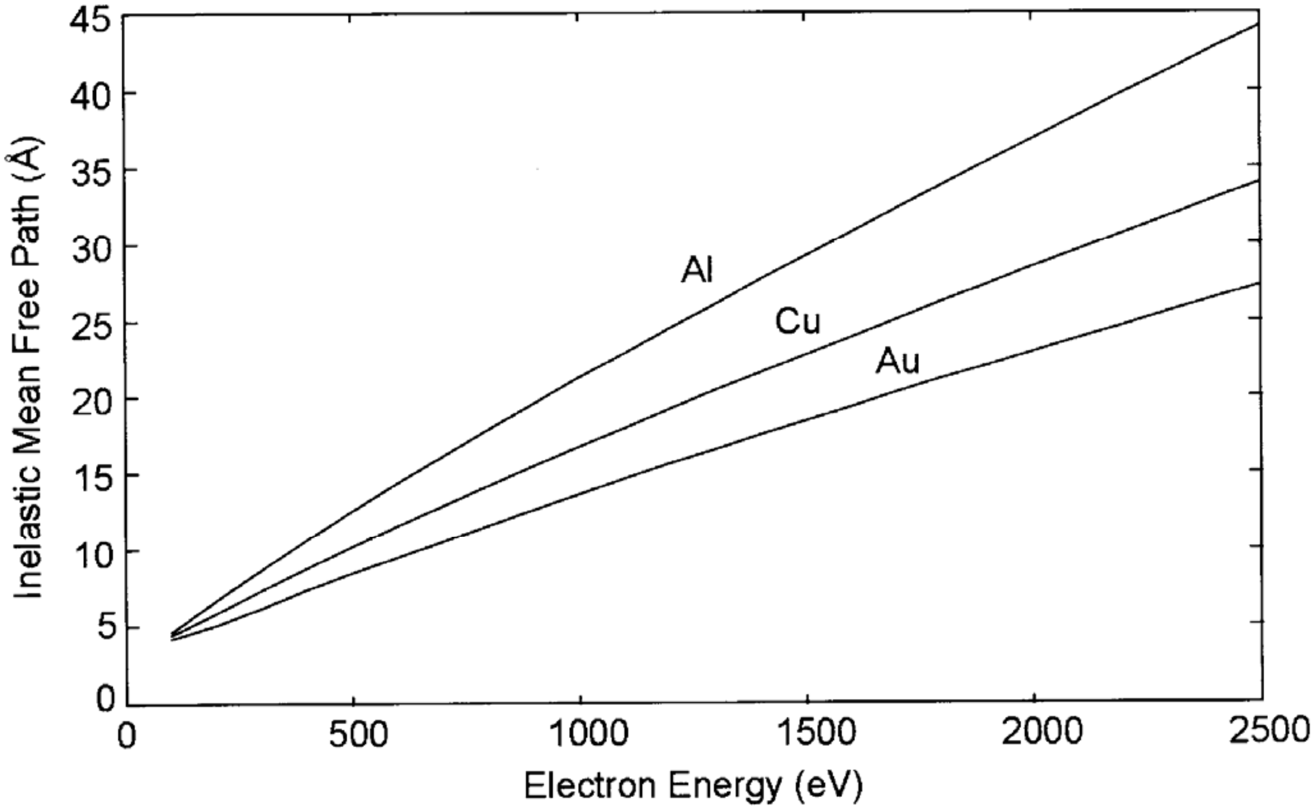
<u>Depth Information and Sputter Rates</u>

Oxide sputter rates 3
Film thickness guide 4
Film thickness dielectrics 5
Common material sputter rates 6
Layered Material Guide 7
Thin Film analysis protocol 11
Preferential Sputtering 15
Sputter rates compound semiconductors and electronic materials 18

From: "Improving surface-analysis methods for characterization of advanced materials by development of standards, reference data, and interlaboratory comparisons" D R Baer, Surface and Interface Analysis 39 (2007) 283

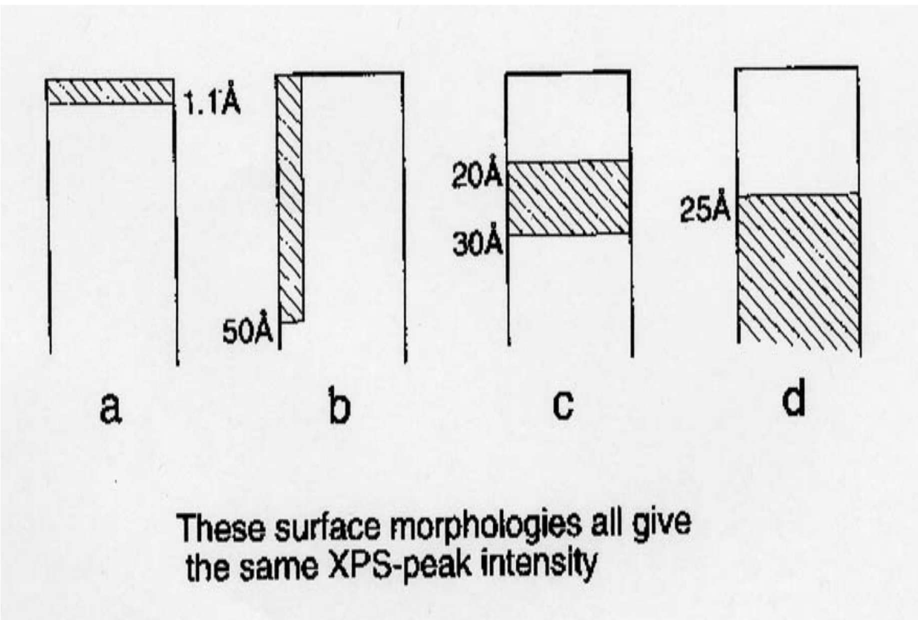
Inelastic Mean Free Paths determine the Information Depth for Auger Electron Spectroscopy and X-ray Photoelectron Spectroscopy.

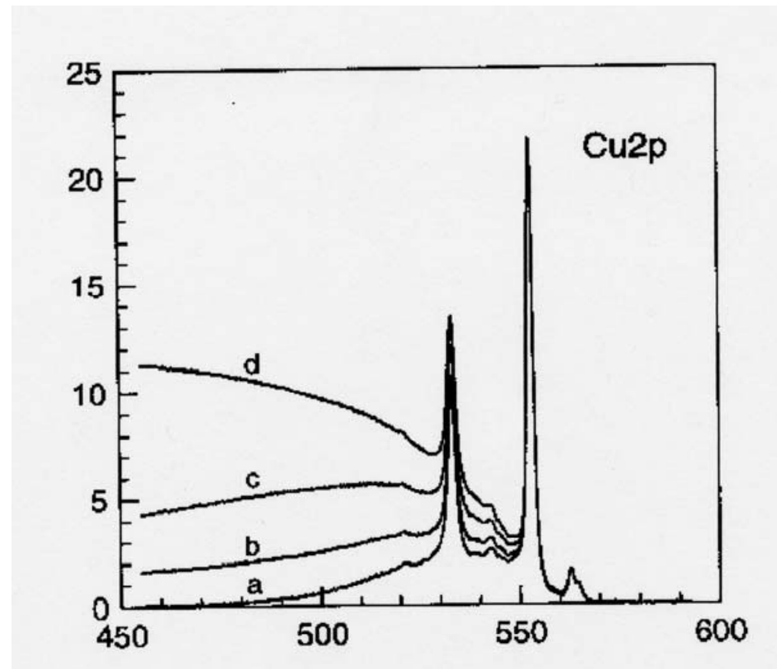
IMFP database www.nist.gov/srd/nist71.htm



Nanometer Distribution of Elements Near a Surface

Non-destructive Depth Information

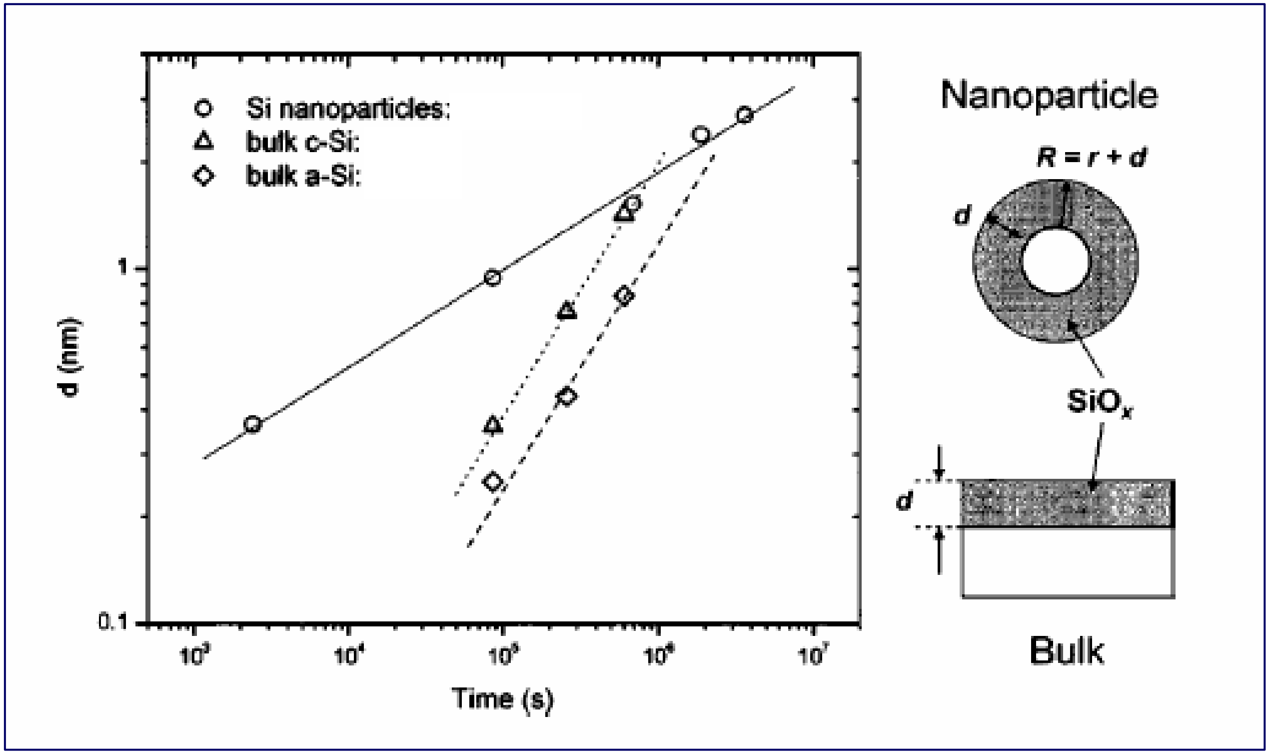




JOURNAL OF APPLIED PHYSICS 97, 024303 (2005)

Room temperature oxidation kinetics of Si nanoparticles in air, determined by x-ray photoelectron spectroscopy

D.-Q. Yang, Jean-Numa Gillet, M. Meunier, and E. Sacher[a)]

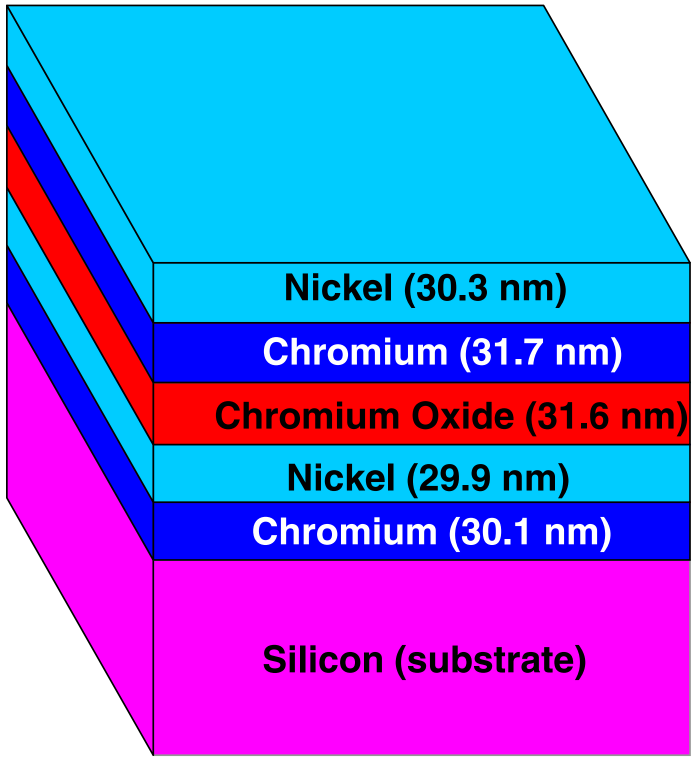


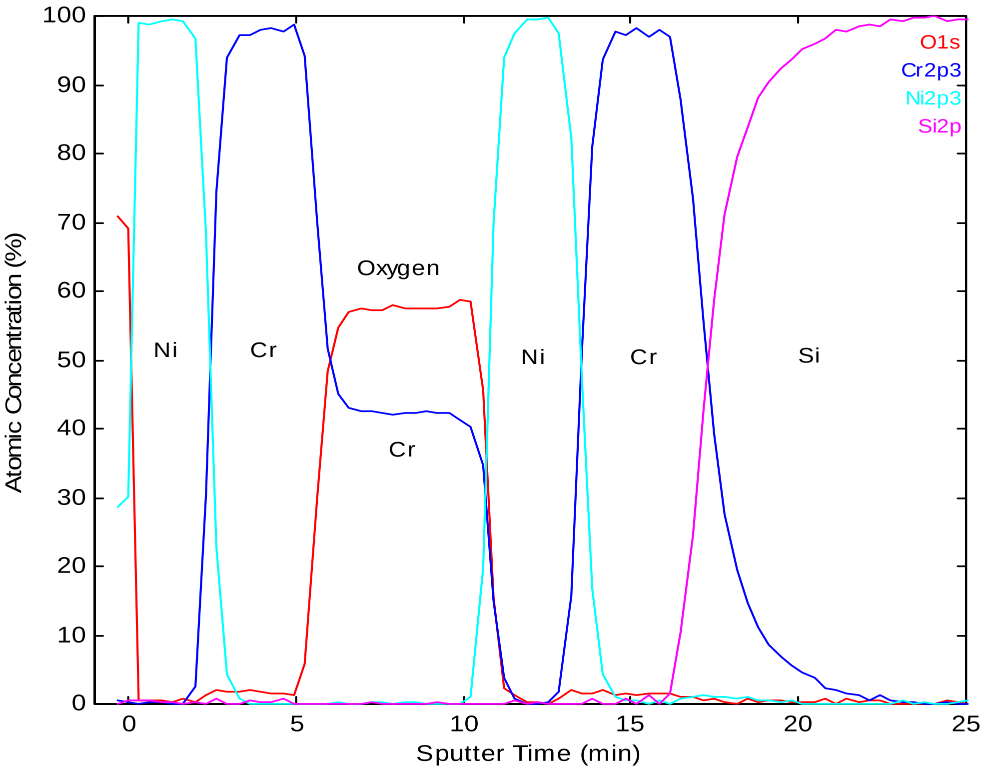
Oxidation of Si spherical nanoparticles deposited on graphite by laser ablation. The oxidation was done at RT in dry air

The oxide thickness was calculated from measurement of the Si from the core and SiO_x from shell

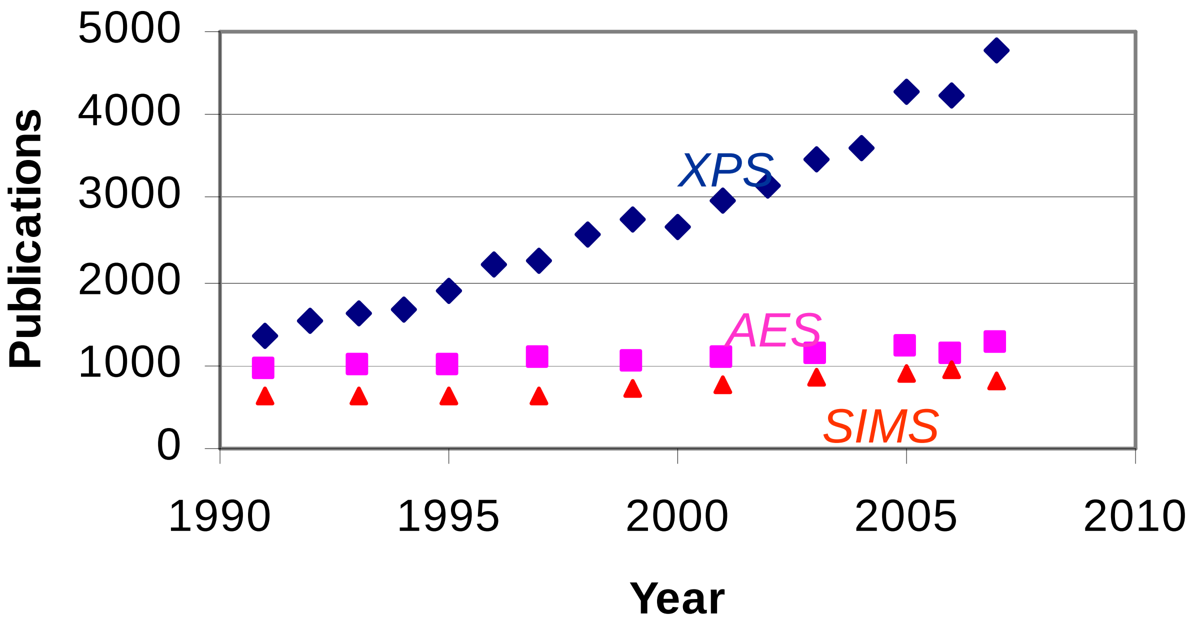
Nanometer-layer information using ion sputtering

XPS Profile Sputter Depth Profile of Multilayer Ni/Cr/CrO/Ni/Cr/Si





Numbers of publications by year with XPS, SIMS or AES as a topic

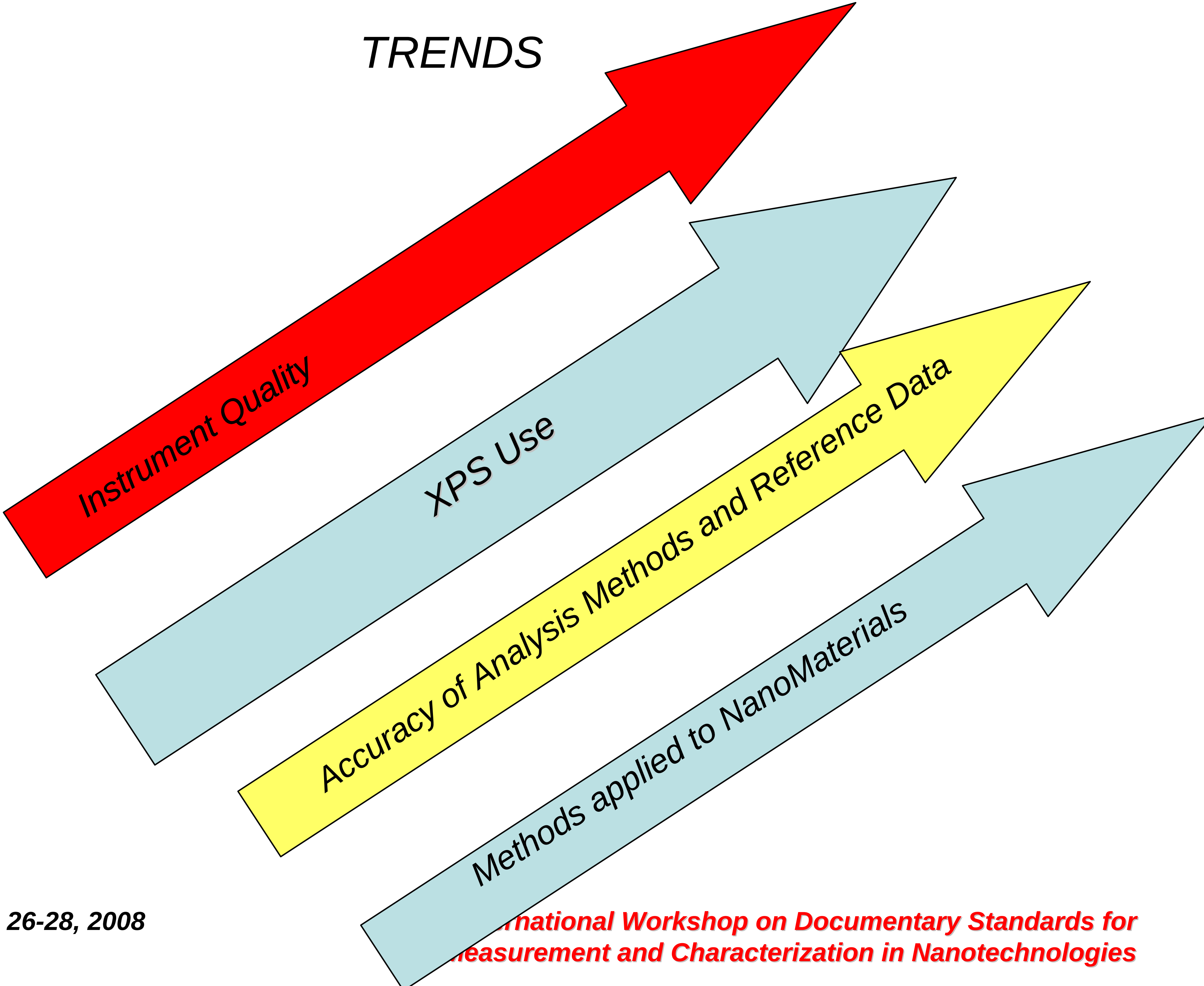


Web of Science topical search:

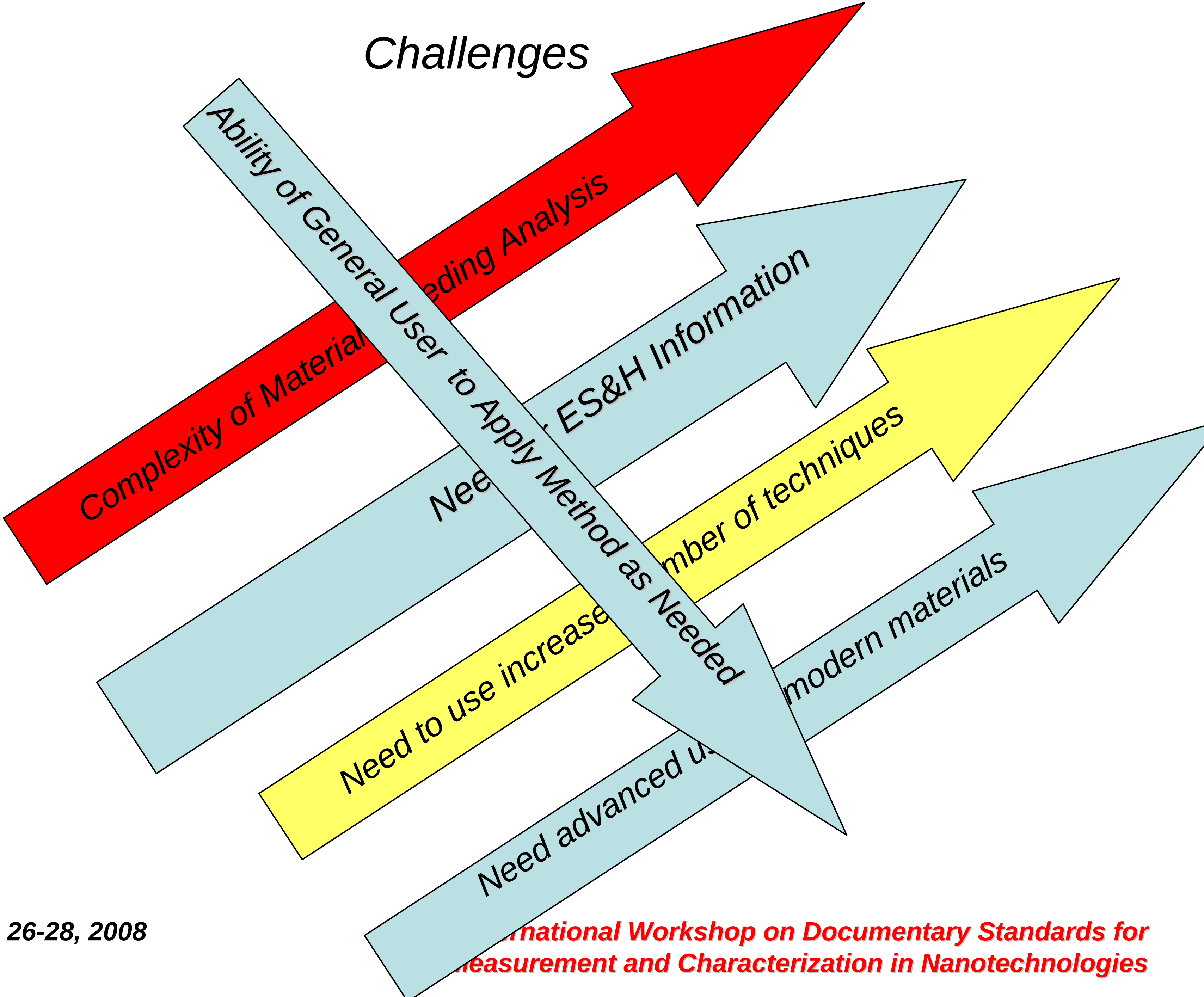
TS=(SIMS or "secondary ion mass spectrometry")
TS=(AES or "Auger electron spectroscopy")
TS=(XPS or "x-ray photoelectron spectroscopy")

Update of graph prepared by Cedric Powell for JVST A 21(2003) S42

TRENDS


Instrument Quality
XPS Use
Accuracy of Analysis Methods and Reference Data
Methods applied to NanoMaterials

Challenges


Complexity of Material
eding Analysis
Nee
ES&H Information
Need to use increase
mber of techniques
Need advanced u
modern materials
Ability of General User to Apply Method as Needed

Technical and Sociological Challenges

- Taking full advantage of what is known
- Extracting information needed about complex and nanostructured materials
  - Consider topology and structure in analysis
  - Complementary information from many techniques
  - Increased environmental and probe sensitivity
- Passing along expert knowledge about application approaches to users who need to use an increasing number of methods in complex ways

USA National Academies Study of Advanced Research Instrumentation

- Instrumentation is increasingly advanced, pushing the boundaries of our science and engineering knowledge and our technologic capabilities…
- The need for particular types of instruments and facilities has broadened, crossing scientific, engineering, and medical disciplines.
- Instruments that were once of interest only to specialists are required by a wide array of scientists to solve critical research problems.

The National Academies Press, 2006

New Type of Standard or Guide

- Protocol or Guide of how experts in specific areas would approach a technical or analysis problem using a surface analysis method. *For example, researchers who study corrosion ask specific questions from XPS spectra. They collect the spectra in some relatively standards ways to answer those questions and process the data to meet the analysis needs.*

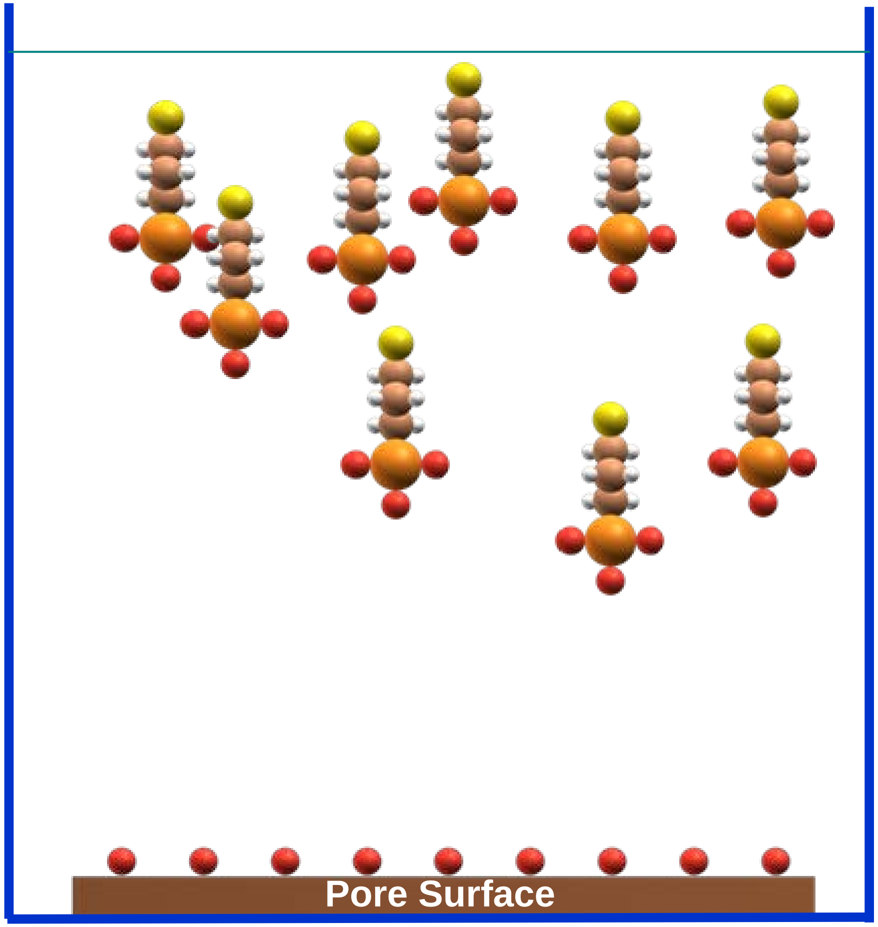
- Having a “consensus” protocol provides guidance and direction to dealing with complex analysis issues and passes on the knowledge of experts.

- Protocol of thin corrosion layers under development. Self Assemble Monolayer Characterization planned.

SAMS Self Assembled Monolayers

Molecular self-assembly

Self-assembly driven by Van der Waals interactions between chains, as well as the interaction between the headgroup and the surface.



"Designing Surface Chemistry in Mesoporous Silica" in "Adsorption on Silica Surfaces"; pp. 665-687, Marcel-Dekker, **2000.**

How to Make Progress?

- Protocol guides should be useful for characterization of nanomaterials, nanostructures, nanoparticles
- Joint working groups TC 229, TC 201 (& others) on **specific** problems/materials
- TC 201 prepare more **general** document(s) on the application of surface analysis to nanomaterials in coordination with TC 229 *(an example will be presented in TC201 presentation)*