Memo CP-D/246

April 15, 1994

To: Distribution

From: H.D. Lemmel

Subject: CPND needs expressed in an IAEA meeting

The IAEA Physics Section held a Consultants' Meeting on the Use of Lower Energy Accelerators for Characterization of Materials in Vienna, 14-17 December 1993. Please, find attached the Final Report of this meeting. The recommendations include a strong request for evaluated charged-particle nuclear reaction data files needed for ion beam analysis for materials research.

Herewith I forward this request to the CPND centers.

While the meeting stated that neutron nuclear data needed for materials research are available with a high level of quality, it is recommended that such data (like the c.p. data) be presented in a user friendly PC format as required for the materials reseach applications.

The papers presented at this meeting will be published as an IAEA-TECDOC.

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Consultants' Meeting on the Use of Low Energy Accelerators for Characterization of Materials

Final Report

IAEA Headquarters

14 - 17 December 1993
Vienna, Austria

Introduction

The use of ion beam analysis (IBA) for materials research was the topic of this Consultants' Meeting (CM). The meeting was a logical continuation of two previous CMs which dealt with the use of low energy accelerators [1, 2]. Among the conclusions reached at these CMs was that a low energy accelerator (200 kV to 5 MV) could be an extremely useful tool for developing countries - as well as in support of IAEA programmes. For example, significant information could be obtained for environmental, biomedical and materials fields and for industrial development areas using these accelerators.

In this report we limit the discussion to the specific field of materials characterization using IBA techniques. These techniques can provide information which can not be obtained by other methods. The IBA techniques themselves, some representative applications and several fields in which IBA is used, are discussed below. This includes materials science techniques and applications for neutron generators, which the IAEA has provided to many member states.

The equally important area of materials modifications using low energy accelerators has not been discussed in this report. It will be the subject of a future CM.

Summary of Discussions

A. The IBA Techniques

Particle Induced X-ray Emission (PIXE).

- Is a widely-used IBA technique for determination of element concentrations above Z=12 in a light matrix. The minimum detection limit is in the range of ppm. It can be considered a relatively fast characterization technique in comparison to other IBA techniques.
- Elemental analysis is the most common application. PIXE can be used with a μm size beam as a surface multi-element mapping technique.
- An advantage is the relatively short time needed for the irradiation of the sample. Also, It can be used as a routine technique (i.e. PIXE need not be changed for different types of samples).

Typical application of PIXE:

- Characterization of major and trace elements in raw materials, alloys, etc.

Rutherford Back Scattering (RBS) & Elastic Recoil Detection Analysis (ERDA).

- The RBS technique enables precision depth profiling of the composition of individual constituents in multi-element structures.
- It can be used for surfaces, surface films and near surface depth distribution analyses.
- Usually it is used together with PIXE as a complementary technique. It can provide the local matrix composition needed for PIXE corrections.
- It is applicable for characterization of light elements.
- Proton and alpha RBS are suitable for characterization of heavy elements in light matrices, such as metallic films on glass. The use of heavy ion projectiles provides high sensitivity for light ions in a heavy matrix.
- The ERDA method is particularly suitable for detection of hydrogen concentrations near surfaces.

Typical application of RBS:

- Measurement of composition and thickness of thin film coatings, corrosion and patina films.
- Determination of diffusion/implantation profiles in semiconductors.

Nuclear Reaction Analysis (NRA, Prompt and Activation).

- NRA is isotope-sensitive, in contrast with other IBA techniques.
- It can be used for bulk analysis.
- It can be used for surface analysis or profiling.
- High beam current is required to perform this technique.
- For properly selected conditions NRA is a powerful technique for materials characterization. However, because the energy of the bombarding projectile and the reaction products to be observed must be selected beforehand, NRA is considered a more difficult characterization technique than the others here mentioned, and virtually impossible to use as a routine analysis method.

Typical application of NRA:

- Thin film activation analysis.

The above techniques may be enhanced by combining them with channelling in single crystals and/or the use of a focused microbeam. These utilizations are now described.

Channelling in crystalline materials

- Aligning the beam with a major plane or axis alters the observed signal by an amount depending on the quality and purity of the crystal lattice. This can be used to investigate crystal defects or the lattice sites of impurity atoms. Applications include the study of crystal damage and annealing following ion implantation (RBS); localization of lattice sites of dopant atoms in crystalline materials (PIXE/RBS); determination of strains in strained layer superlattices (RBS).
- Channelling requires the additional provision of a goniometer in the target chamber

Typical applications of microPIXE:

- Diffusion profiles in polymers
- Mapping metallization patterns in devices
- Composition homogeneity of synthetic materials/ceramics

Typical applications of microRBS:

- Mapping the thickness of surface films
- 3D mapping of heavy inclusions in a light matrix.

Focused Beam Analysis (Scanning Particle Microscopy, SPM)

- Use of a focused beam allows spatial information to be obtained from the sample. This is a major enhancement to the quality of the data - allowing, for example, 2D elemental mapping; the selection of representative regions for analysis; the identification and avoidance of regions of contamination
- The combination of simultaneous PIXE, RBS and other techniques with scanned microbeam has become known as *Nuclear Microscopy.*
- The additional equipment for producing a scanned micrometer diameter beam is relatively complex (but commercially available); however, beams of ~ 25 μm diameter can be produced using relatively simple apparatus (e.g. pinholes, standard quadrupole doublet).

- Additional analytical techniques can be used with a very low current (approx. 2000 particles per second) microbeam. These are:
  - **Scanning Transmission Ion Microscopy (STIM)**

    This measures the energy loss of ions transmitted through the sample to map density or thickness variations (e.g. mapping of metallization patterns on devices, mapping of precipitates in semiconductors materials). STIM can also be combined with Computer Tomography (CT) to obtain three-dimensional images of structures.
  - **Channelled Scanning Transmission Ion Microscopy (CSTIM)**

    The combination of STIM and Channelling allows the direct imaging of crystal defects in "bulk" (up to 50 μm thick) crystals. For example: direct imaging of dislocations at mismatched interfaces.
  - **Ion-Beam-Induced Charge (IBIC) / Single Event Upset (SEU)**

    The charges induced by single ions in the junctions of the operational devices can be used to map the active regions and investigate changes under different circuit conditions. Changes of state induced by single ions in operating logic devices can be used to map radiation hardness (caused by SEUs) and follow the propagation of errors in complex circuits.
  - **Fabrication of high aspect ratio structures**

    Poly-methyl methacrylate (PMMA- an organic glass) etch resists can be exposed to MeV ion microbeams and high aspect ratio structures can be formed (e.g. channels 1 μm wide and 60 μm deep). This could enhance the production of high-device-density masks for X-ray lithography or microstructures).

Neutron based techniques

- The techniques based on 14 MeV neutrons produced from the D-T reaction with low voltage accelerators have the following main advantages: fast and nondestructive, no matrix problems, applicable for small and bulk samples. By using activation and/or prompt radiation methods the elements can be determined in any matrices. The continuous and pulsed mode operations of fast neutron generators can be applied also for the investigation of neutron irradiation effects in solids, using a fluence of approximately 10^{12} - 10^{13} n/cm^2.
- The combination of the sealed-tube accelerator with the associated particle method or using nanosecond pulsed beam with time-gating at the detector, render possible the three dimensional elemental analysis of solids and the elastic scattering analysis of light elements (C, N, O) by back-scattered fast neutrons at the resonance energies. The development of the Pulse Height Response Spectrometer has increased the field of applications of small neutron generators in materials research to include even extremely large samples.
- The D-D neutrons produced from low voltage accelerators have serious limi-

tations in materials research.

Typical applications of fast neutrons in materials technology are:

- Determination of light element concentrations in bulk samples.
- Measurements of total H and He in different metals by quadrupole mass spectrometry (QMS).
- Studies on the tritium breeding and outgassing efficiency for different compounds and alloys.
- Investigations of the release of foreign and lattice atoms by diffusion and evaporation from metals and alloys.
- Studies of the effect of 14 MeV neutron irradiation on semiconductor electronic components, integrated circuits, semiconductor detectors, and electro-optical components.

B. Typical fields of applications in materials technology.

In this section, we present a representative list of the fields to which IBA techniques are making an important contribution.

Metals and Alloys

- Corrosion and wear
- Surface coatings
- Stoichiometry / impurities
- Interface characterization

Semiconductors

- Impurities
- Implantation / diffusion profiles
- Crystal defects / damage
- Device response.

Polymers

- Diffusion into polymer surfaces
- Catalysis residues
- Inclusions in thin films

Glasses / Ceramics

- Surface phenomena
- Stoichiometry

Recommendations

1- Because of the increasing importance of the materials industry to national economies, this Consultants' Meeting strongly recommends to the Agency to promote the use of low energy accelerators for materials characterization among the member states. We recommend that the present level of Agency involvement in this field be increased. This involvement occurs through the CRPs of the Research and Isotopes Department and the national, regional and interregional technical cooperation projects of the Technical Cooperation Department.

2- For existing accelerators, the CM feels that their use can be best optimized through bilateral agreements or regional TC projects.

3- Because PIXE is probably the most versatile, multi-element, fast, quantitative characterization technique, it is recommended that a training course on the use and applications of the PIXE technique should be organized. The emphasis of this course should be on the practical side.

4- The CM notes the high level of quality and availability of neutron nuclear data and the equivalent lack for charged particle nuclear data. Therefore we recommend that the IAEA should encourage and promote efforts in the nuclear data field towards the establishment of experimental and evaluated charged particle nuclear data files relevant to accelerator-based IBA techniques. It is also recommended that these data be presented in a 'user friendly' PC format.

References

1.- Consultants' Meeting on low energy accelerators in elemental analysis, Chiang Mai, Thailand, 25-29 March 1991. IAEA Internal Report

2.- Consultants' Meeting on present status of low energy accelerators. Debrecen, Hungary. June 1992. IAEA Internal Report.

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