

D10 2 Survey Report Final

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AHEAD WP10

D 10.2 Survey Report Final

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1. SUBJECT

This document reports the outputs from the application survey campaign that has been carried out in the frame of the AHEAD WP10 “Technology Innovation” activities.

The main content is a commented list of the techniques and application fields which have been identified during the study phase, both for ground applications and for space applications.

The present issue has been updated for the final release of the document in Q1 2019.

1.1. STRUCTURE OF THE DOCUMENT

The document is structured as follows:

Chapter 3 describes the general scenario related to WP10 (Technology Innovation)

Chapter 4 identifies the main technologies under development in the frame of the AHEAD project and gives a selection of many experimental techniques related to these technologies

Chapter 5 gives a survey of applications (ground & space) based on the techniques described in chapter 4 and the rationale for the activities follow-up.

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1.2. ACRONYMS LIST

ADC	Analog to Digital Converter
AHEAD	integrated Activities for the High Energy Astrophysics Domain
CryoAC	Cryogenic Anti-Coincidence
CT	X-ray Computed Tomography
DAC	Digital to Analog Converter
DR	Dynamic Range
EC	European Commission
EDXRF	Energy Dispersive X Ray Fluorescence
ESA	European Space Agency
EU	European Union
EXAFS	Extended X-ray Absorption Fine Structure
FEE	Front-End Electronics
FDM	Frequency Division Multiplexing
FLL	Flux Locked Loop
IBA	Ion Beam Analysis
ICD	Interface Control Document
IFU	Integral Field Unit
IXS	Inelastic X-ray Scattering
NDT	Non Destructive Testing
NRIXS	Non Resonant Inelastic X-ray Scattering
PCB	Printed Circuit Board
PIXE	Particle (Proton) Induced X-ray Emission
PIGE	Particle (Proton) Induced Gamma-ray Emission
P/L	Payload
QC	Quality Control
RIXS	Resonant Inelastic X-ray Scattering
SAXS	Small Angle X-ray Scattering
SDC	Silicon Drift Chamber
SDD	Silicon Drift Detector
SIXS	Soft Inelastic X-ray Scattering
SQUID	Superconducting Quantum Interference Device
SRON	Space Research Organization of Nederland
SpW	SpaceWire
TAS	Thales Alenia Space
TAS-I	Thales Alenia Space-Italia
TBC	To Be Confirmed
TBD	To Be Defined
TBV	To Be Verified
TBW	To Be Written
TES	Transition Edge Superconductor
TDM	Time Division Multiplexing
TM/TC	TeleMetry and TeleCommand
TRXRF	Total Reflection X Ray Fluorescence
WDXRF	Wavelength Dispersive X Ray Fluorescence
WP	Work Package
XANES	X-ray Absorption Near Edge Spectroscopy
XAS	X-ray Absorption Spectroscopy
XES	X-ray Emission Spectroscopy
XFEL	X-ray Free Electron Laser
XPS	X-ray Photoelectron Spectroscopy
XRD	X-ray Diffractometry
XRF	X-ray Fluorescence
RRR	X-Ray Radiography
XRS	X-ray Raman Scattering (mode of IXS)

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2. APPLICABLE AND REFERENCE DOCUMENTS

2.1. APPLICABLE DOCUMENTS

AD1: AHEAD GRANT AGREEMENT
Grant Agreement number: 654215 — AHEAD — H2020-INFRAIA-2014-2015

2.2. REFERENCE DOCUMENTS

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"Broadband Ultrahigh-Resolution Spectroscopy of Particle-Induced X Rays: Extending the Limits of Nondestructive Analysis"
 PHYSICAL REVIEW APPLIED 6, 024002 (2016)
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 CIRP Annals - Manufacturing Technology 63 (2014) 655–677
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"Recent Developments in X-ray Imaging Technology"
 Lawrence Livermore National Laboratory report LLNL-TR-587512, June 12, 2012
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 Supercond. Sci. Technol. 28 (2015) 084003 (36pp)

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3. INTRODUCTION

AHEAD (integrated Activities for the High Energy Astrophysics Domain) is a collaborative research project carried on by a consortium of 26 institutions from 16 countries within the Horizon 2020 initiative funded by the European Commission.

The focus of the AHEAD research project is to coordinate the advancements in the field of High Energy Astrophysics at many levels, including the development of enabling technologies for state-of-art instrumentation to be used for the observation of astrophysical targets.

The reference frame for the project is the ESA Cosmic Vision L-class ATHENA mission, a large part of the research effort is put in the development of a focal plane array of TES microcalorimeters used as x-ray detectors. The ultimate goal of the project is anyway wider and foresee enabling technologies also in other fields like gamma-ray instruments.

The AHEAD project also includes a dedicated work package (WP10) devoted to technology innovation with the aim to explore the transfer of technologies developed in the AHEAD project to other applications relevant to the high-tech industrial market, both in the space domain and for “ground” applications.

The WP10 tasks include an application survey to identify those applications that could benefit from AHEAD advancements: the following chapters describe the results of this survey.

4. TECHNIQUES IDENTIFICATION PROCESS

The survey of the applications of the technologies under development in AHEAD requires a preliminary identification of these technologies and of the experimental techniques that could possibly benefit from these developments.

From the proposal documentation [AD1] we can identify the following technologies under development in the frame of the AHEAD project:

- TES-based microcalorimeters for x-ray spectroscopy (WP6)
- Specialized electronics for the readout of an array of TES using SQUIDs (WP6)
- Cryo Harness (WP6)
- X-ray optics (WP8)
- Optical blocking filters (WP6)
- Background control (including particle-specialized microcalorimeters for AC) (WP6/WP7)
- Gamma-ray detectors (WP9)
- calibration procedures for x-ray / soft gamma rays instrumentation (WP4)

The set of above listed technologies may found a natural use in those applications based on x-ray or gamma-ray techniques that are already in use nowadays and can be improved by the availability of higher energy resolution and/or more efficient detectors systems.

Moreover some other emerging or new techniques are identified which could be improved or made feasible by the technologies developed in AHEAD.

Most of the x-ray techniques used nowadays are based on solid state detectors (mostly Silicon, Si-Li and in some cases Germanium or less common materials like e.g. GaAs).

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A table comparing the characteristic features of the most common detectors with the expected improvements from AHEAD is reported herebelow:

Parameter	Typical values	Potential improvement from AHEAD developments (qualitative)	Remarks
Energy range	0.1 keV to > 100 keV	Efficiency at higher energies may be improved using microcalorimeters with proper absorber selection	
Energy resolution	100 - 150 eV @ 5.9 keV (SDC, with moderate cooling)	< 2eV @ 5.9 keV with TES microcalorimeters (requiring sub-K cooling)	
Spatial resolution	10 µm (CCD) to 500µm (pixellated detector) to tens of mm (large area detectors)	Comparable to pixellated detectors	
Timing resolution	Few µs	Not likely to compete due to the inherently slow thermal response	
Signal intensity/rate	1-10 ksp/s / pixel	Not likely to compete	
Collecting area	Very wide range,	Design dependent	
Special thermal requirements	Detector often cooled below 270 K for better resolution	Detector must be cooled to << 1 K	

Table 4.1: typical x-ray detector characteristics w.r.t. potential improvement from AHEAD

It should be noted that the various research fields using x-ray have developed an increasing large number of different techniques, [REF3, REF4, REF9], most often bearing overlapping definitions and with similar general description so that obtaining a clear view of the state-of-art is not an easy task.

As a first general classification we can divide the x-ray techniques in the following types:

- a) direct imaging (e.g. radiography, CT)
- b) diffraction imaging (e.g. XRD)
- c) direct spectra collection (e.g. XAS, XES)
- d) energy-loss spectra collection from scattering experiment (e.g. IXS, XRS)
- e) direct imaging + spectrometry (e.g. IFU)

The first two classes require mostly photon counting detectors so that the most relevant potential innovation from AHEAD could be those related to:

- increased detector efficiency (useful to reduce dose on samples)
- background reduction (through more selective energy discrimination)

On the other hand the last three classes strictly require spectroscopic detectors so that in these cases the most relevant innovation from AHEAD should be in the field of:



- increased energy resolution

Increased energy resolution and increased detector efficiency for x-ray are two of the key features of the TES microcalorimeters developed in AHEAD.

IFU (point “e” above) will target both good imaging and high energy resolution, fundamental for diffuse or faint sources.

Also x-ray optics with increased spatial resolution is within the AHEAD goals, we see then that the connection between AHEAD and these techniques worth an in-depth investigation.

The following chapters reports a resume of the experimental techniques identified so far which are related to the AHEAD main research topics and AHEAD technologies.

Each technique is briefly reminded and a set of characteristic parameters is given to be compared with related performances of the target technologies for AHEAD.

Applications, both for use on ground and for space, descending from the techniques identified in this chapter are dealt for in chapter 5.

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4.1. X-RAY RADIOGRAPHY (XRR)

This is by far the most common x-ray technique, having a long time heritage in the field of material inspection and medical imaging.

This technique requires an x-ray source in the 30 to 60 keV (typ) range and an imaging (or at least position sensing) detector sensitive to the same energy range.

The basic operation is to illuminate the sample with a x-ray beam while recording the amount of photons passing through it.

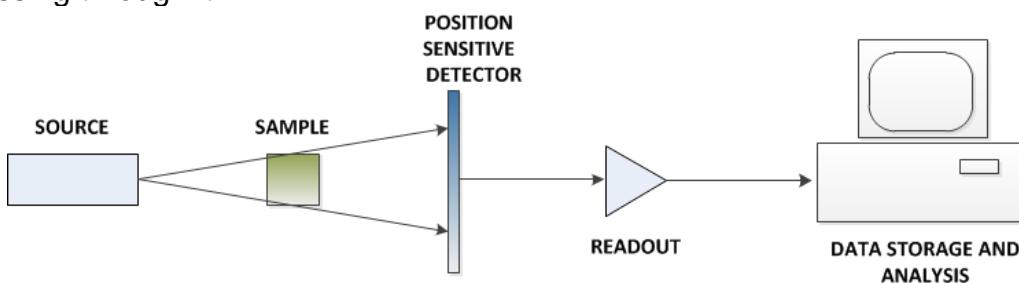


Fig. 4.1.1 sketch of a typical set up for X-ray radiography

In a simplified model the number of photon per unit time (flux) passing through the sample is described by the Beer-Lambert law:

$$I(z) = I_0 \cdot e^{-\mu \cdot z}$$

Where I_0 is the flux of incoming photons and z is the depth within the sample.

The absorption coefficient μ [m^{-1}] depends on the sample material and on the photon energy in a way expressed by the Bragg-Pierce law:

$$\mu \approx \rho \cdot Z^4 / (h \cdot v)^3$$

Where Z is the atomic number of the atoms in the sample and $h \cdot v$ is the energy of the x-ray photons so that the absorption coefficient is higher for dense and high-Z materials and decreases with the photon energy. It is also common in literature to express this dependence in terms of the mass attenuation coefficient μ/ρ which is weighted w.r.t. the material density ρ .

Either simple shadow recording or the use of a focusing optic may be implemented in the different set up following the specific needs of the user.

In its simpler implementation the primary end-products of this technique are images based on the different absorption characteristic of the various sections of the sample.

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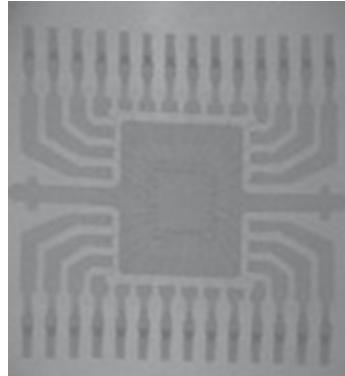


Fig. 4.1.2 example radiography of the interior of an electronic device

More advanced implementation are also in common use:

- Dual energy
- “Colour” X-ray radiography

These techniques are based on the capability to discriminate the photon energy in the images (either acting on the source or on the detector) so that further computer-based processing can be used to produce images where different materials are evidenced.

The idea under the dual energy technique is that the attenuation of an X-ray beam passing through a target is a combination of two photon-matter interaction processes: photo-electric effect and Compton scattering. The level of attenuation coming from one or the other process depends on the photon energy and the material Z so that measuring at two distinct energies in principle allows disentangling the attenuation into two these two components, so permitting to discriminate between two materials of different Z.

The Color X-ray technique is an extension of the dual energy under development in these years taking advantage from the availability of photon counting detectors with energy measurement capability.

Main uses

- medical diagnostic (including dental)
- veterinary diagnostic
- nondestructive inspection (materials, welding/castings, assemblies, archeology research ...)
- security (person/luggage inspection etc.)

Discussion on potential improvement from AHEAD developments:

Being tailored for low-rate (faint) sources the microcalorimeters studied in the AHEAD project requires high efficiency to not waste photons. The microcalorimeters design could be optimized for efficiency by proper selection of the absorber materials: this could be exploited to set up a low-dose x-ray radiography so to allow observation of delicate samples or specimen in material science or biology and medicine.

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On the other hand scaling down the spatial resolution to what is presently obtained by conventional detectors (e.g. $\sim 10\mu\text{m}$ with CCDs) will pose a challenge in terms of readout multiplexing to a degree probably higher than what is tackled in AHEAD.

It should be noted that the recently emerging “microwave multiplexing” technique may overcome these limits, anyway this specific technique is not included in the AHEAD developments.

4.1.1. X-ray Computerized Tomography (CT)

This technique may be seen as an evolution of x-ray radiography where the sample is illuminated in a scanning mode from many directions and the obtained pattern of shadowgrams is analysed by SW to obtain a 2D picture of the interior of the sample along a cross-section plane or a “slice”.

3D graphic SW can be used to align many CT “slices” to obtain a 3D picture of the internal structure of the sample.

The source energy is usually in the 10 keV to 150 keV range, up to 450 keV range in industrial applications, depending on the materials to be inspected, and the detectors works in the same energy range.

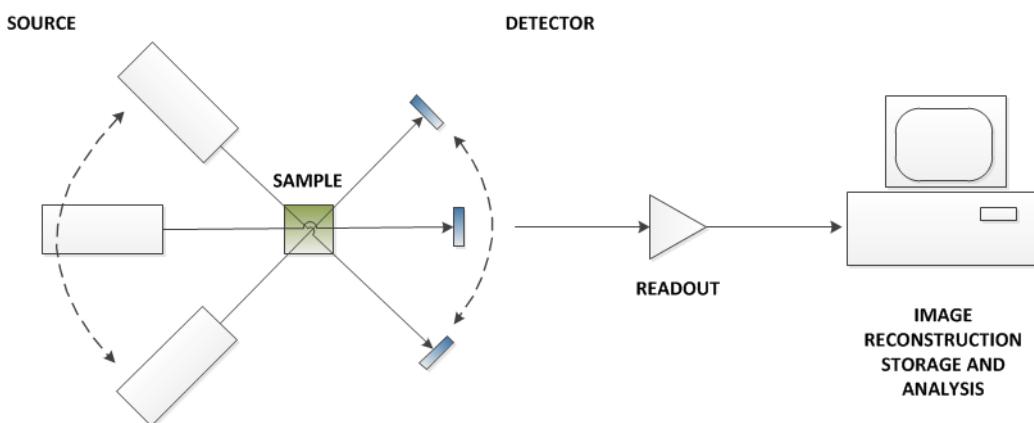


Fig. 4.1.1.1 sketch of a typical set up for X-ray CT

The source is aimed at the sample along different angles while a set of detectors, or a single detector rotated together with the source, collect the x-ray passing through the sample itself.

Alternative set up foresee fixed source-detector alignment and a rotating sample. Helical scanning may also be implemented for sample which have an elongated shape as is the case of the human body for clinical CT.

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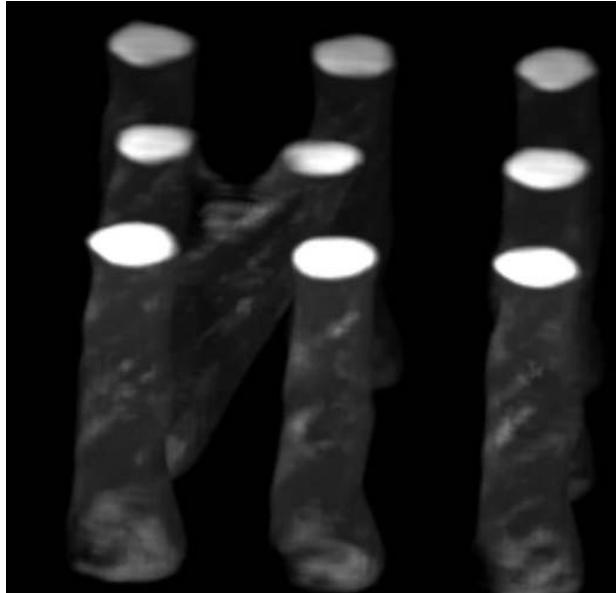


Fig. 4.1.1.2 example of CT inspection of a column-grid assembly showing a defect

Main uses

- medical diagnostic
- nondestructive inspection. (materials, assemblies, archeology research ...)
- dimensional control/metrology [REF18]

Discussion on potential improvement from AHEAD developments:

Similar to what observed for radiography, anyway in this case the source is usually more an hard x-ray so that the microcalorimeter design should include a tailored absorber.

One possible advantage of higher energy resolution obtainable from TES microcalorimeters is the possibility to reduce the background disturbances (e.g. from fluorescence) exploiting the better energy discrimination provided by the TES.

The disadvantage of the TES arrays in terms of spatial resolution may be less critical here as the images are obtained from the scanning pattern of the source rather than from a position sensitive detector as in the case of simple radiography.

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4.1.2. Positron Emission Tomography (PET)

This technique is based on the detection of the coincidence emission of two 511 keV photons from the annihilation of a positron emitted by a proper “tracer” substance which has been purposely distributed into the explored volume.

The detection of a couple of coincident photons, which are emitted at 180° directions by momentum conservation law, provides a line-of-sight of the annihilation site.

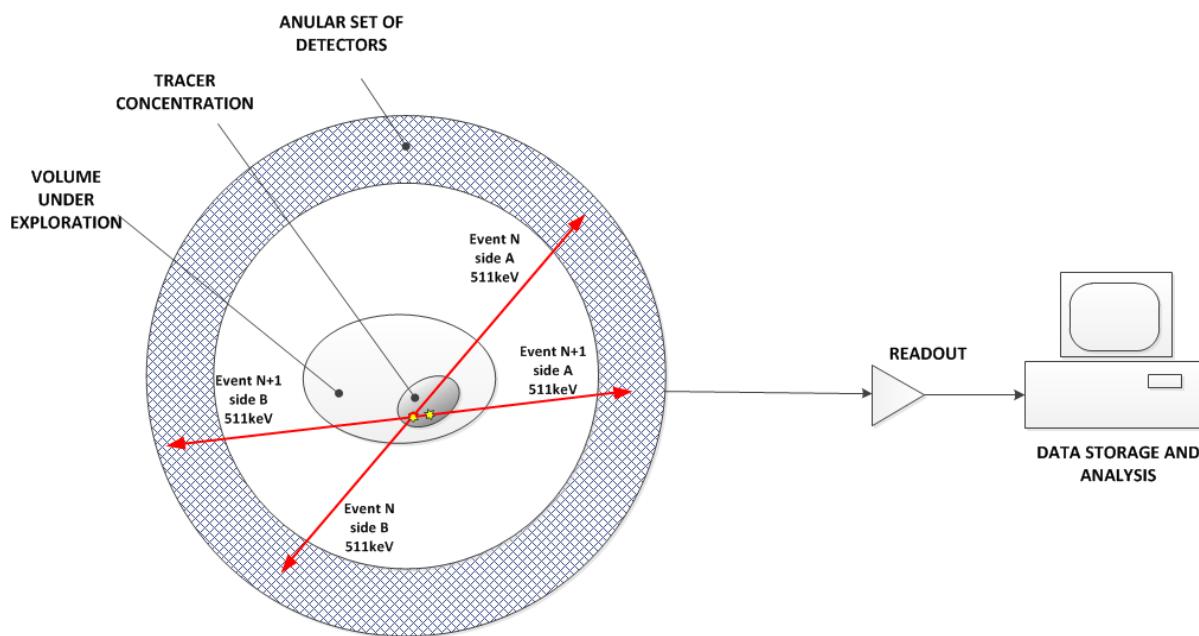


Fig. 4.1.2.1 sketch of a typical set up for PET

Accumulating many such events it is possible to reconstruct the distribution of the tracer inside the explored volume

The detection system is often arranged as an annular set of individual detectors with coincidence circuitry to trigger on the opposite elements across a diameter.

This technique is mostly used in medicine or veterinary diagnostics where it provides an important mean to visualize FUNCTIONAL structures like organs or tissues where the specific chosen tracer accumulates by biological/metabolic processes. This is different from radiography and CT imaging systems which provides less specific structural images.

Main uses

Medicine or veterinary for image-diagnostic and identification of metabolic activity centers

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Discussion on potential improvement from AHEAD developments:

It is a technique based on gamma-ray and quick-responding detectors (coincidence) so that TES are likely not providing advantages.

Gamma detectors with high speed response studied in WP9 may be considered.

4.2. X-RAY SPECTROSCOPY

This is a well-established experimental techniques which relies either on energy-dispersive set-up followed by position-sensitive detectors or on detectors capable to stop the x-ray photons and analyse their energy content.

Two main class of experiments can be identified:

- X-ray Absorption Spectroscopy (XAS)
- X-ray Emission Spectroscopy (XES)

4.2.1. XAS

The XAS technique requires an intense and fine-tunable monochromatic source, usually a synchrotron x-ray source, in the 1 to 10 keV range with a detector in the same range.

The basic operation is to sweep the energy of the incident beam while recording the amount of photons passing through the sample.

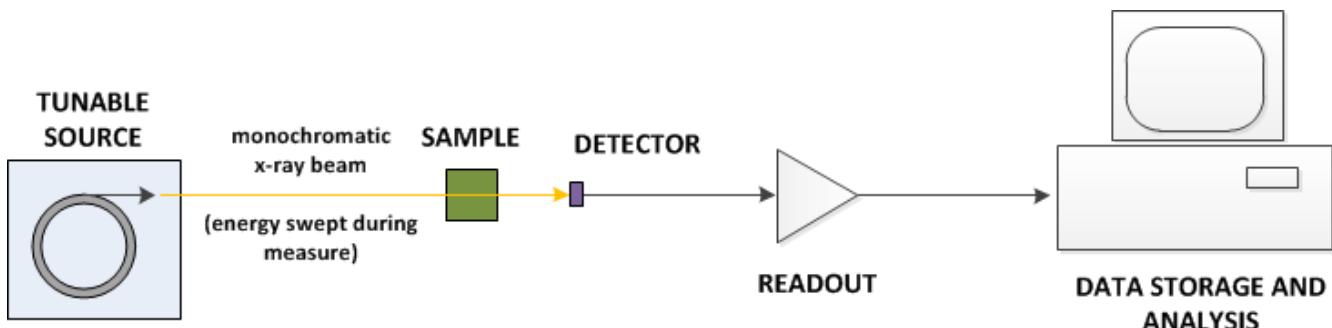


Fig. 4.2.1.1: simplified sketch of a XAS set-up

Works better for atomic species with $Z > 22$

The primary end-product of this technique is a profile of the absorption coefficient $\mu(E)$ reflecting the energy levels of the unoccupied valence states of the sample atoms.

The absorption coefficient is characterized by a general decreasing profile interrupted by some sudden increases (absorption edges) at sharp energy values.

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The energy of the various absorption edges strictly depends from the atomic elements involved, while the fine structure of the edge profile near the edge and at energy just above are related to their chemical and physical states which can be studied by dedicated measurement techniques (see XANES and EXAFS subchapters).

A sketch of a generic XAS spectrum around an absorption edge with indication of the common nomenclature of the most important features is given in the following figure.

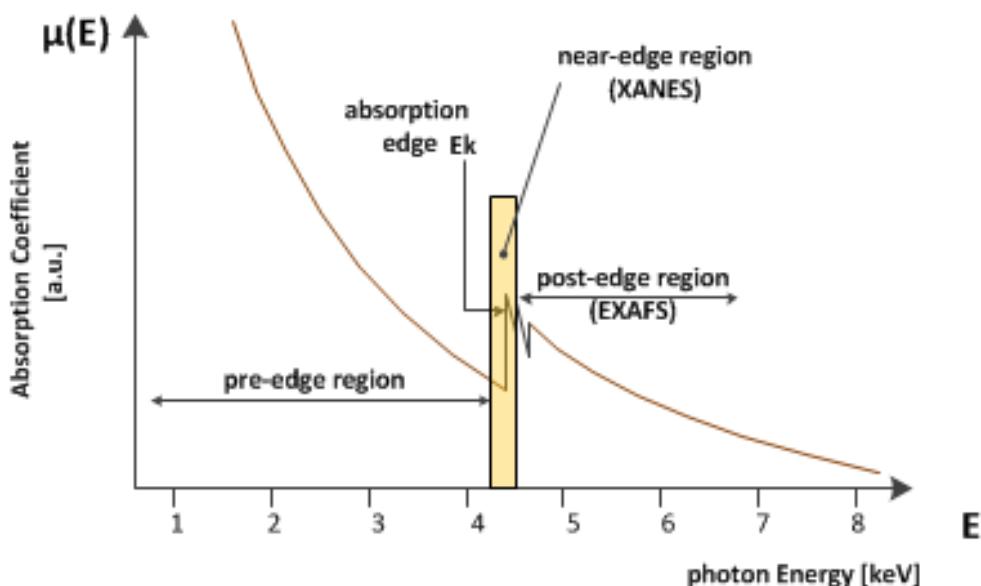


Fig. 4.2.2: sketch of a XAS spectrum

The energy resolution in the edge identification can be as low as some eV depending on the narrow tuning capability of the monochromatic source.

Main uses

Elements identification

Discussion on potential improvement from AHEAD developments:

The energy resolution in XAS comes from the monochromaticity and tunability of the source so that TES detector does not add value in this respect but they can be of advantage in terms of better signal over background (e.g. induced fluorescence).

The TES may also allow higher efficiency than traditional Si detectors when working around higher x-ray ranges.

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4.2.1.1. X-ray Absorption Near Edge Spectroscopy (XANES)

A XAS variant known as XANES works just around the absorption edges of the interesting elements and is often used together with x-ray microscopy to provide accurate maps of target elements and their chemical state.

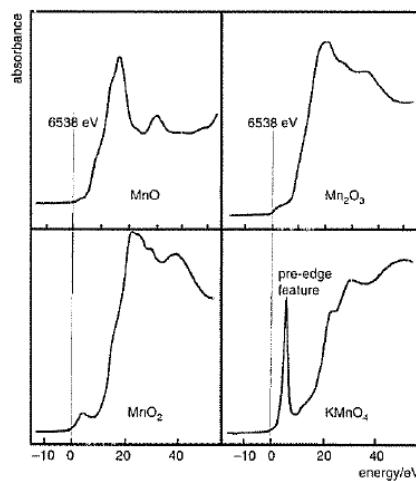


Fig. 4.2.1.1.1: example of XANES spectrum of various Mn compounds (energy given as difference w.r.t. reference a 6.538 keV line)

Main uses

Elements identification

Chemical composition analysis

Discussion on potential improvement from AHEAD developments:

Same general consideration as for XAS

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4.2.1.2. Extended X-ray Absorption Fine Structure (EXAFS)

EXAFS is another variant of XAS which focuses on the “ripple” features in the “extended” region just above the absorption edge to get information about the interatomic distances in the probed sample. These information give hints on the structure of the sample at atomic level.

The theory behind this technique is based on the interference between the photoelectrons generated by the x-ray excitation and the wavefunctions of the neighbour atoms which lead to minima and maxima ripples in post-edge absorption profile. The computation is carried out by Fourier transformation of the difference signal between the measured spectrum profile and an idealized “ripple-less” profile to extract the interatomic distances.

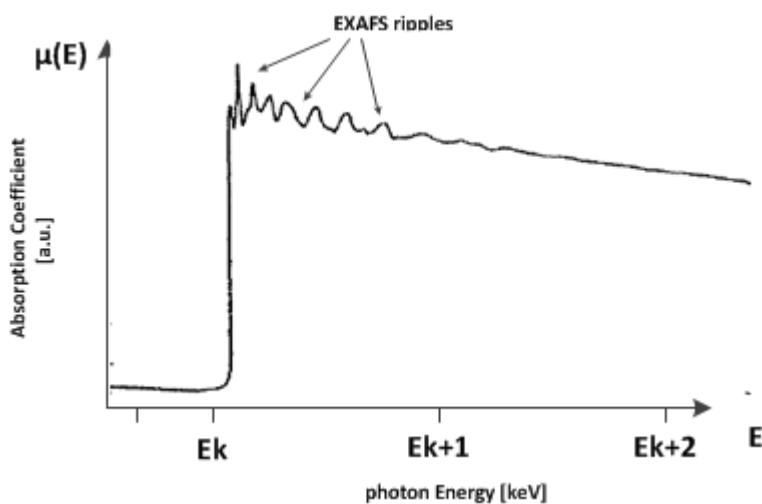


Fig. 4.2.1.2.1: example of EXAFS spectrum

Main uses

Information on the structure of the sample (interatomic distances)

Discussion on potential improvement from AHEAD developments:
Same general consideration as for XAS

OPEN

4.2.1.3. XES / XRF

XES is a general term which encompasses all the techniques which rely on the analysis of the x-ray photons emitted by the sample when its atomic electron systems relax after an excitation provided by a proper source capable to create an empty core state in the electron distribution. In general any XEF set up requires an intense excitation source with energy higher than the studied states, 1 to 15 keV is normally used with an energy sensitive detector working in the same energy range. A synchrotron x-ray source is ideal but also broadband is applicable, in some application the source may be a radioisotope.

The basic operation is to illuminate the sample with the excitation radiation while recording the amount and energy of the emitted photons.

In particular the XRF (X-ray Fluorescence) technique is based on the spectroscopy analysis of the secondary x-rays emitted by a material sample stimulated by an energetic source (usually x-ray at higher energies).

The secondary x-rays emitted by the sample are characteristics of the elements composing it so that the collected spectra may be used to investigate the sample composition [REF3].

The general set up include at least the following elements

- a source to excite the atoms in the sample
- an “optical” section (at least a collimator, possibly a focusing system)
- an x-ray detector
- an electronic chain (front-end + bias +back end for analysis)

The primary end-product of this technique is an energy spectrum reflecting the energy levels of occupied valence states, the energy resolution can be as low as 100-150 eV at detector level but can be narrower in some set up which uses a swept tunable source.

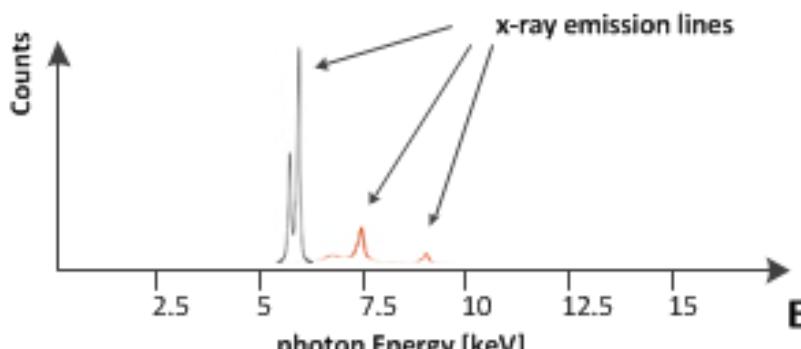


Fig. 4.2.2.1: sketch of a XES spectrum

A classification can be made on XRF systems following their operation:

- Energy Dispersive X-Ray Fluorescence (EDXRF)
- Wavelength Dispersive X-Ray Fluorescence (WDXRF)
- Total Reflection X-Ray Fluorescence (TRXRF)



4.2.1.4. Energy Dispersive X-Ray Fluorescence (EDXRF)

In EDXRF fluorescence emission is analysed by a spectroscopy detector connected to a multichannel analyser so that all spectral lines are acquired in parallel.

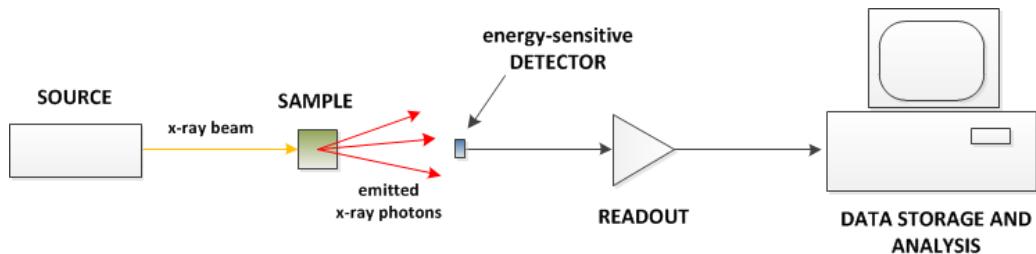


Fig. 4.2.2.1.1: sketch of a EDXRF spectrum

The primary end-product of this technique is an energy spectrum.

The typical energy range is in the few keV and energy resolution is in the order of 100-500 eV.

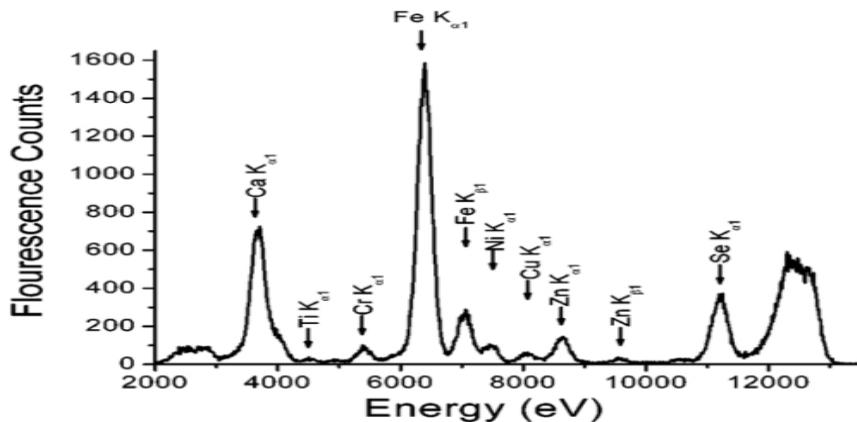


Fig. 4.2.2.1.2 sketch of a EDXRF spectrum

Main uses

Chemical research, identification of elements

Is a much versatile technique for non-destructive elements identification from Sodium (Na) to Uranium (U) where the sample may be powder, liquid or solid.

Used in hazardous substance detection and alloy composition identification.

Discussion on potential improvement from AHEAD developments:

Some benefits could come from TES resolution at eV level [REF1], also TES microcalorimeters may be designed to be more efficient than usual Si detectors in the harder x-ray range.

These features can be very useful in experiments with low intensity source beams, either due to source limitations or when analysing radiation sensitive samples.

In recent times TES microcalorimeters arrays are increasingly used in XES applications, in particular with ultra-fast sources or in electron microscopes and accelerators beamlines.

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4.2.1.5. Wavelength Dispersive X-Ray Fluorescence (WDXRS)

In **WDXRF** the fluorescence emission is dispersed by a proper crystal (monochromator) so that each wavelength has its own direction, a simple photon counting detector is then swept through the various dispersion angles to record the intensity of emission at that direction.

In some applications a 1D array of photon counting detector is placed across the swept angle to collect all wavelength at the same time.

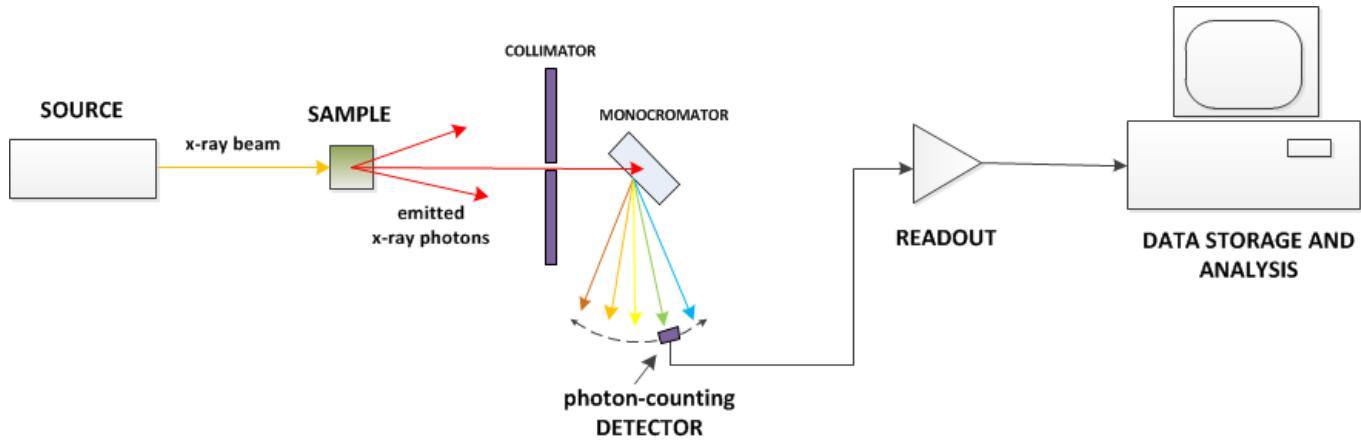


Fig. 4.2.2.2.1 sketch of a WDXRF set up

The WDXRF techniques allow a better achievable energy resolution w.r.t. EDXRF but is limited to soft x-ray with a practical limit around 2.5 keV where WDXRF can achieve resolutions down to 5 eV to 20 eV. On the other hand the additional optical components of a WDXRF system (monochromator and collimators) lead to a reduced efficiency, i.e. longer time to collect enough data or use of higher intensity sources, and higher costs.

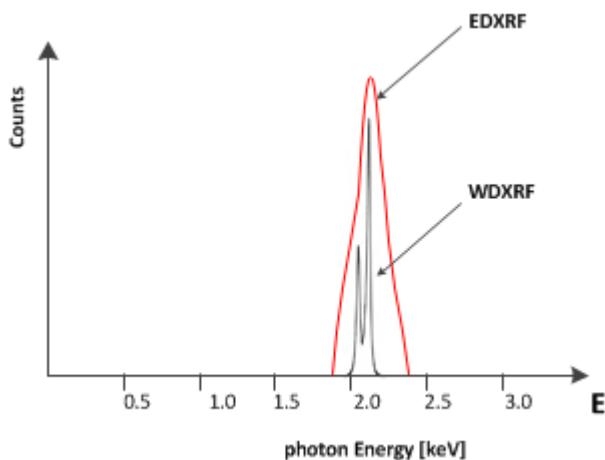


Fig. 4.3.2.1 qualitative sketch of a WDXRF spectrum compared with a EDXRF

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Main uses

nondestructive technique used in many industries for high accurate elemental analysis
capable to give information of bonding states
used in geoscience
used in environmental analysis
used in food safety
used analysing archaeological artefacts.

Discussion on potential improvement from AHEAD developments:

TES microcalorimeters likely would not add value to this technique w.r.t. standard detectors (typ Si-Li) as their much better energy resolution is not relevant for these dispersive set up.
A possible plus could be the potential higher efficiency in some applications.
Basically if a TES detector was available then EDXRF would be preferred to WDXRF in most cases thanks to the TES high efficiency combined with good energy resolution and easier handling and alignment of the related set-up.

4.2.1.6. Total Reflection X-Ray Fluorescence (TRXRF)

In **TRXRF** the stimulating beam is applied at very low incident angles ($< 0.1^\circ$) so to get XRF from only a small thickness surface layer that can so be analysed independently from the substrate. The excitation efficiency of the sample is maximized by this arrangement which also reduces both the adsorption and the scattering of the exciting beam within the sample so leading to lower background noise and overall higher sensitivity.

This technique is useful to analyse really small amount of sample materials and it is somehow easier to operate in the field w.r.t. other methods.

It is similar to EDXRF in most of the other aspects.

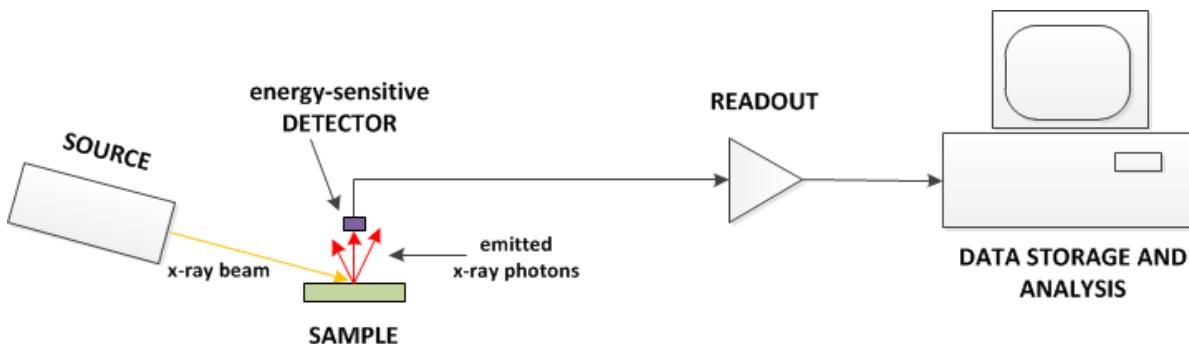


Fig. 4.2.2.3.1 sketch of a TRXRF set up

Main uses

Chemical analysis at trace level
Analysis of metal impurities in chemical reagents
Water/Air environmental analysis

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Discussion on potential improvement from AHEAD developments:

Same as per EDXRF

4.3. ELASTIC X-RAY SCATTERING

Under the general term of Elastic X-ray scattering fall a set of techniques (XRD in primis) which rely on the detection of the x-ray photons that are elastically scattered from the sample, i.e. they are deviated from the original direction maintaining the same energy.

4.3.1. X-Ray Diffractometry (XRD / TXRD)

This technique is based on the detection of x-ray from a known collimated and monochromatic x-ray source which have been diffracted by the lattices at atomic level when passing through a material sample.

The diffracted x-rays from the sample are directed into a pattern which reflects the internal lattice organization in terms of geometry and spacing. The analysis of the XRD pattern can be used to investigate the sample crystalline structure and in some cases composition.

The general set up include at least the following elements

- a collimated monochromatic source to excite the atoms in the sample
- an “optical” section (at least a collimator, possibly a focusing system)
- a x-ray detector
- an electronic chain (front-end + bias +back end for analysis)

A classification can be made on XRD system following their operation:

- Standard XRD
- Time-Resolved X-Ray Diffraction (TXRD) (100 fs level to “freeze” lattice movements)

The following table gives a quick resume of these methods.

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Technique	Sub technique	Source characteristics	Sample characteristics	Detector characteristics	Analysis products
XRD X-Ray Diffractometry	XRD Basic operation: Probe the sample with a monochromatic and collimated (" few μm) source few 1000 cps rate 5-20 keV range	Requires a monochromatic and collimated (" few μm) source few 1000 cps rate 5-20 keV range	solid, powder	1D or 2D Imaging detector may be just counting often based on CCD	Diffraction pattern reconstruction with few μm resolution Typ Energy Range: 5 to 20 keV
	TXRD Time-resolved XRD Basic operation: Like above but taking data in "bunches" of <1 ps duration (either by pulsing the source or by fast-shutter the detector)	Case 1 pulsed at 100 fs timescale Case2 standard	solid	Case 1 x-ray detector Case2 ultra-fast sub-ps (streak camera)	Evolution of sample structure under stimuli at fs scale

Table 4.3.1.1: XRD and TXRD characteristics

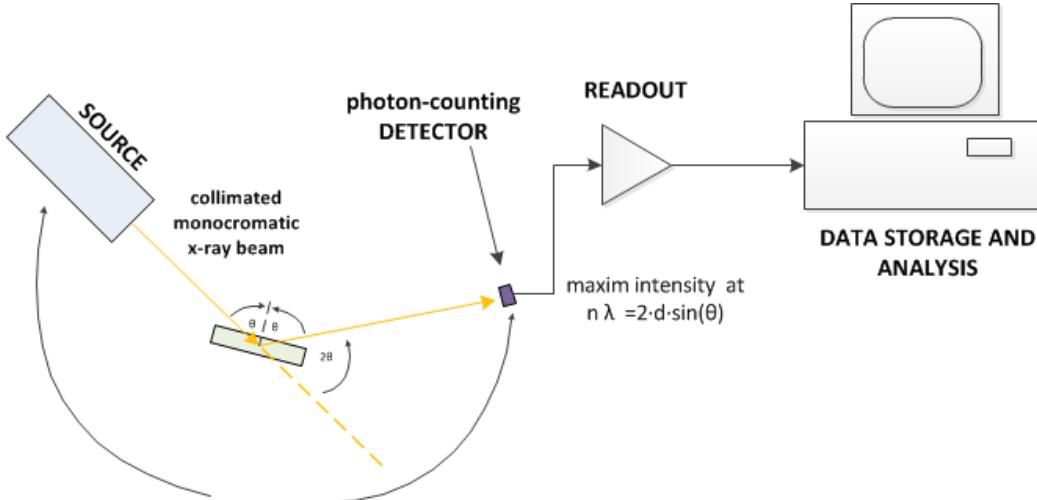


Fig. 4.3.1.1 sketch of a typical XRD set up: note that source, sample and detector are moved together so to maintain the detector always aligned with the reflection angle θ while this is swept in an operating range between 0° to 90° .

The primary end-product of this technique is a diagram showing the recorded counts intensity vs. the θ angle: being known the x-ray wavelength λ this information may be used to derive the value d of the distance between atomic layer in the sample crystals.

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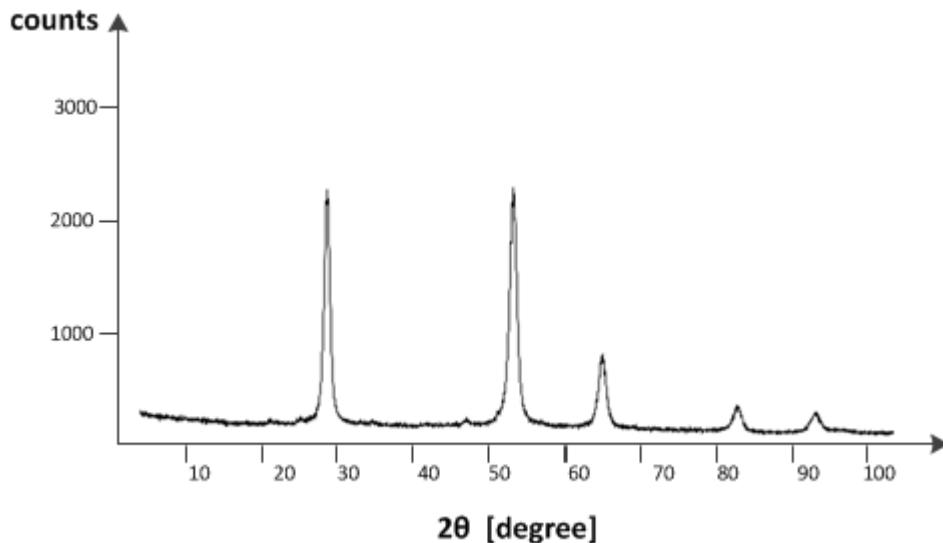


Fig. 4.3.1.2 sketch of a XRD diffraction diagram

Main uses

Mineral identification

Crystal structure characterization (e.g. in pharmaceutical industry)

Microelectronics wafer defects inspection

Discussion on potential improvement from AHEAD developments:

Being a technique based on photon counting detectors the TES microcalorimeters likely can not add value standard detectors. To be explored if the increased energy resolution of TES microcalorimeters may improve background rejection.

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4.3.2. Small Angle X-ray Scattering (SAXS)

This technique is based on the detection of X-ray scattered by the sample at very small angles w.r.t. the incident beam, it is used for characterization of surfaces or objects in the nm scale. The typical set-up foresees a monochromatic collimated source impinging on the sample either in transparency mode (for thin samples) or in grazing mode (surfaces) while the scattered pattern is collected by a suitable 2D position-sensitive detector on-axis with the beam with proper beam-stop to get rid of the source beam.

Main uses

Protein structure characterization
Surfaces characterization

Discussion on potential improvement from AHEAD developments:

Being a technique based on photon counting detectors and moreover requiring high 2D spatial resolution the TES microcalorimeters likely can not add value w.r.t. the use of standard detectors.

4.4. INELASTIC X-RAY SCATTERING (IXS)

Inelastic X-Ray Scattering (IXS) includes a broad family of techniques based on the analysis of x-ray photons coming from a known source that have been scattered by interaction with the sample matter in an “inelastic” way so giving part of their energy to the sample itself (or in some cases taking energy from the sample if it was in an excited state).

The scattered x-rays from the sample have a direction which is related to the direction of the incoming radiation and depending on the particular interaction that has taken place in the sample [REF4].

A general characteristic of this family of techniques is the low efficiency of the process, so that high intensity sources are needed to provide results in a reasonable time, usually the experiments are carried out at a synchrotron light beamline or at a XFEL facility.

These experimental set up are usually large (in the order of some meters, neglecting the dimension of the source facility).

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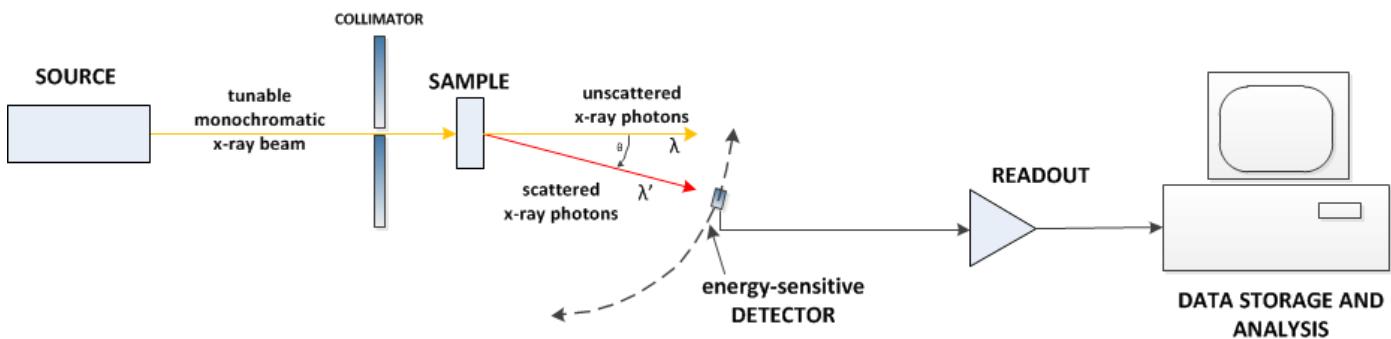


Fig. 4.4.1 sketch of a general x-ray scattering set-up
(may vary depending on the experimental details)

Depending on the experimental details, namely the energy range and the particular process which is providing the scattering, there are many possible experimental set up that we try to classify in the following table:

Experimental Set Up	Typical source energy	Interaction process	Achievable energy resolution
SIXS (Soft Inelastic X-Ray scattering)	0.1-2 keV (tunable)	outer electronic shells	10 meV
NRIKS (Non-Resonant Inelastic X-ray Scattering)	10 -15 keV	Inner core electrons	
XRS (X-ray Raman Scattering)	10-15 keV (tunable)	inner core electrons	< 1 eV
RIXS (Resonant Inelastic X-Ray scattering)	2-15 keV (tunable)	Inner core electrons	100 meV
Compton scattering	50-100 keV (tunable)	inner electronic shell of Hi-Z elements (only efficient in the hard X-ray range)	

Note1: some degree of source tunability is often required to this kind of experiments

Note2: position-sensitive detectors are required in most of these experiments to measure the scattering angle, these are normally provided as an array of individual detectors placed in an arc around the sample or sometimes by a single detector on a rotating structure that is scanned across the scattering range during the experiment.

4.4.1. Soft X-Ray scattering (SIXS)

This technique requires a monochromatic and collimated source of X-ray in the 0.1 to 2 keV (typ) range.

The basic operation is to sweep the energy of the incident beam while recording the amount of photons scattered by the sample at different angles.

The primary end-product of this technique is the energy-loss spectra of the scattered photons in an energy range between 10 meV to 1 keV eV and resolution down to 10 meV at 500 eV.

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This technique is suited to the investigation of energy levels in low-Z atomic species and in the study of binding energies of molecules.

Discussion on potential improvement from AHEAD developments:

TES optimized for very low energies may improve the efficiency and possibly resolution through a more accurate determination of the scattered photon energy.

4.4.2. Non Resonant X-ray scattering (NRIXS)

This technique requires a x-ray source with energy high enough to kick-off an inner core electron to a continuum ionized state.

The basic operation is to adjust the source energy high enough and to detect both scattered photons and the photon emitted by the atomic shell reorganization processes.

The primary end-product of this technique is the x-ray spectrum in the range 0.1 to 10 keV (typ) for various scattering angles of both scattered and emitted photons which can be used to assess the energy distribution of excited states in the valence and conduction band of the sample material.

This technique is suited to investigations in the field of material science.

Discussion on potential improvement from AHEAD developments:

TES optimized for very low energies may improve the efficiency and possibly resolution through a more accurate determination of the scattered photon energy.

4.4.3. X-Ray Raman Scattering (XRS)

This technique is based on the detection of x-ray from a known monochromatic x-ray source which have been scattered at large angles by an inner (core) electron of the atoms composing the material sample [REF19].

This technique allows the use of high energy x-ray probing in the investigation on light (low Z) materials. This gives advantages in terms of better penetration in the bulk of the sample or when operating in air or liquid environment.

Indeed for the low-Z elements, which have correspondingly low-energy absorption edges, the XAS (X-Ray Absorption Spectroscopy) technique become inefficient due to the low energy (so low penetration) photons involved.

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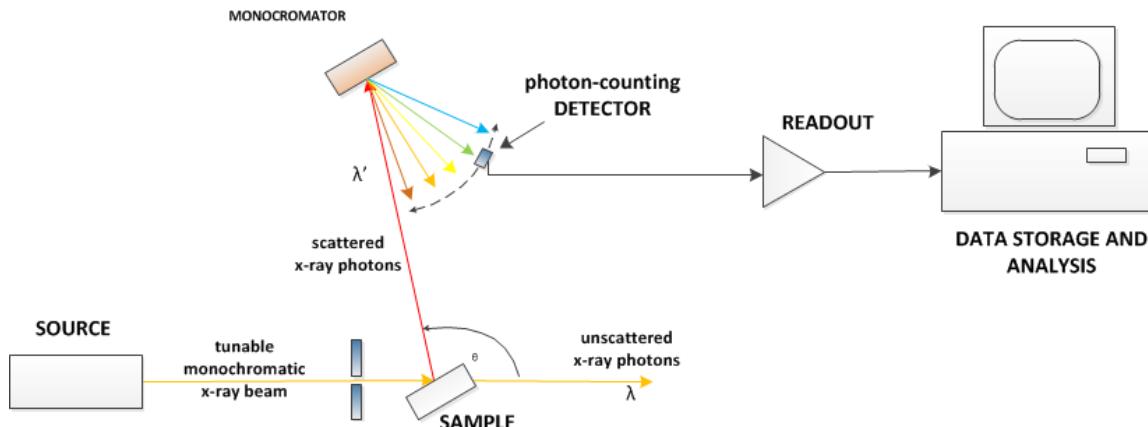


Fig. 4.4.3.1 sketch of a typical XRS set-up using a wavelength dispersive scheme

The basic operation is to sweep the source energy so to kick core electrons into an higher level unoccupied state and to record the energy and angle of scattered photons. Photons scattered at large angles falls in the soft x-ray and are commonly analyzed using a wavelength dispersive scheme.

The primary end-product of this technique is the energy-loss spectrum of scattered photons in the range between 0.05 to 1 keV (typ) with energy resolution of few eV at 500 eV.

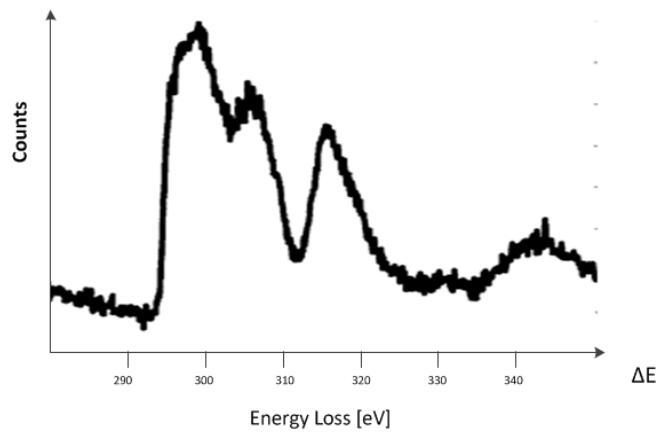


Fig. 4.4.3.2 sketch of a XRS spectrum of C

Main uses

Same uses as XAS and related techniques, optimized for low-Z materials in bulk solid, liquid or air test environment.

Discussion on potential improvement from AHEAD developments:

TES optimized for low energies used in an energy sensitive scheme may improve the efficiency and possibly resolution trough a more accurate determination of the scattered photon energy.

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4.4.4. Resonant Inelastic X-Ray Scattering (RIXS)

This technique is based on the detection of the changes in energy, momentum and polarization (in some cases) of the scattered photons which are related to the excitation states of the target under irradiation.

The energy of the source beam is chosen to “resonate” with one of the atomic transition of the sample so to increase the scattering cross section.

It is a technique which is being developed in the these years due to the availability of intense x-ray beamlines like XFELs and improved instrumentation for the detection of the low-energy scattered photons. A comprehensive description can be found in [REF 20].

The basic set-up is composed by a source which should be collimated and highly monochromatic (to sub-eV passband) tunable plus focusing optics and a spectrometer assembly.

The spectrometer takes different implementation following the hard or soft x-ray to be analyzed basically using a grazing incidence grating for soft x-ray or a Bragg crystal analyzer for hard x-rays. Due to the geometrical constraints of the scattering process these spectrometer set-up are usually quite large, the detectors being mounted onto scanning arms having a length in the order of a few meters.

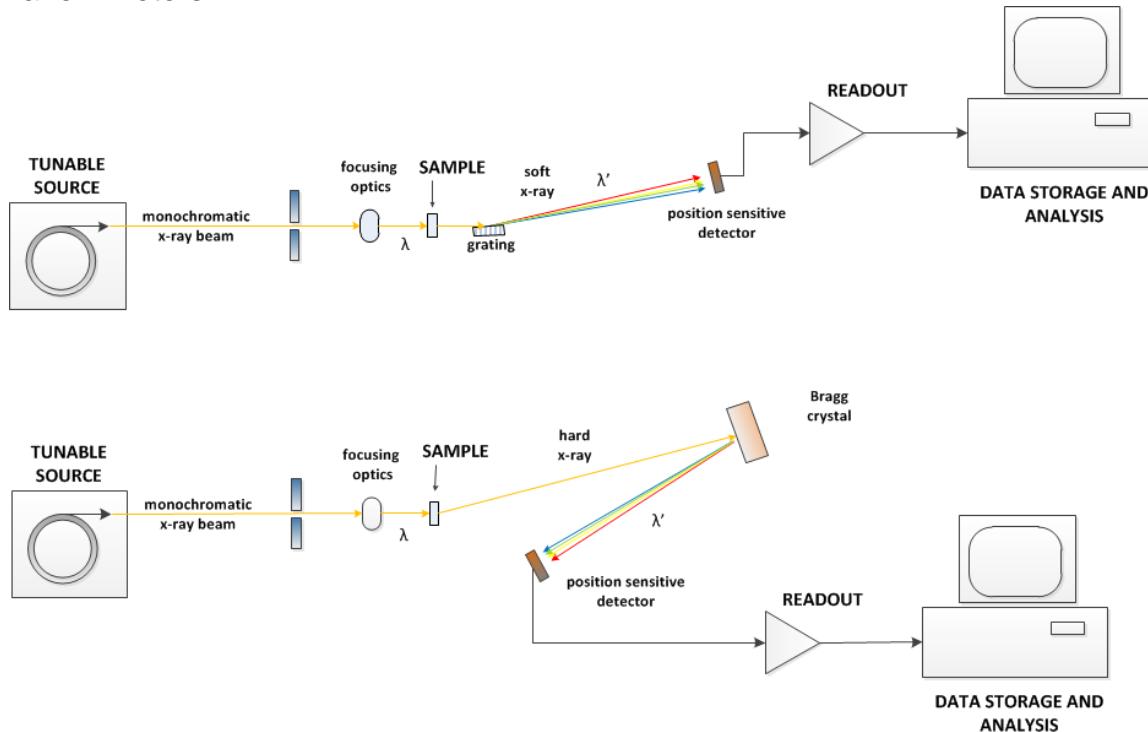


Fig. 4.4.4.1: simplified RIXS set-up for soft x-ray target radiation (upper sketch) and hard x-ray target radiation (lower sketch)

The basic operation is to tune the source energy on the particular transition to be studied so to kick core electrons into a higher level unoccupied states and to record the energy and angle

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(and in some cases polarization) of the scattered photons following the reorganization of the shell structure. This process is polarization-dependent and also sensitive to the magnetic moment of the sample, it is well suited to study superconducting materials.

The primary end-product of this technique is the energy loss spectrum of scattered photons in the energy range 0.1 to 1 keV with sub-eV resolution. From the knowledge of the energy of the incident photons (strictly monochromatic) it is then possible to obtain the energy profile of the transition at the scattering centre.

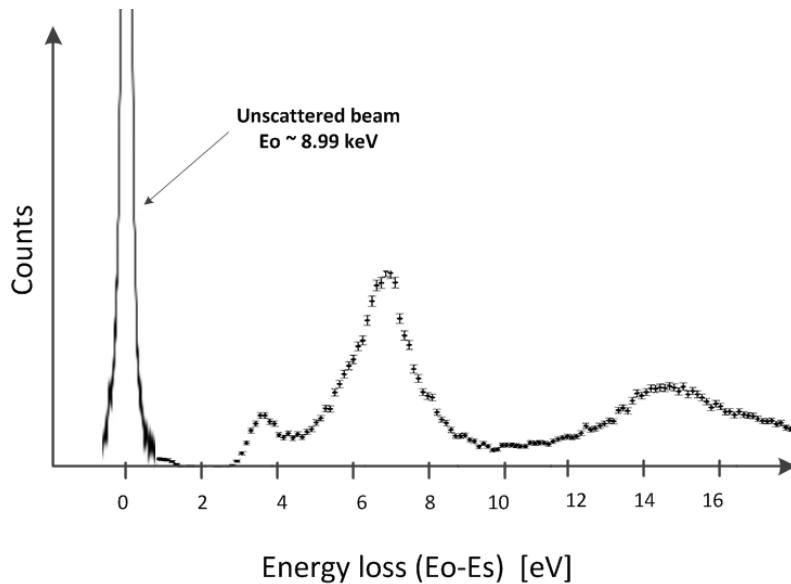


Fig. 4.4.4.2 sketch of a RIXS spectrum (hard x-ray type)

Discussion on potential improvement from AHEAD developments:

The value of this technique is the achievable energy resolution in the sub-eV range so that even TES detectors can hardly provide any improvement, unless for lower-end systems which could benefit from a more compact spectrometer section based on TES optimized for very low energies.

4.4.5. Compton Scattering

This effect is related to the scattering of incoming photons by loosely bound electrons in the outer shells of the target atoms.

This process is efficient only at higher energies w.r.t. the main band studied in AHEAD, and is rather suited for experiments like those studied in WP9 (Gamma Ray Experiments).

Anyway the recent availability of extremely brilliant x-ray sources like XFELs makes this technique usable also for x-ray in certain circumstances.

The basic operation is to illuminate the sample with collimated hard X-rays or gamma rays and to record the spectrum of scattered photons as a function of the scattered angle.





The ruling law which relates the energy (E_{ph}) of the scattered photon with that of the photons of incoming radiation ($E = hc/\lambda$) is described by the Compton's expression:

$$\frac{1}{E_{ph}} - \frac{1}{E} = \frac{1}{m_e c^2} (1 - \cos(\theta))$$

Where m_e is the electron mass and c is the speed of light.

Note that the Compton interaction being with the outer electrons layers only it is not dependent from the target atomic number or density etc.

From the knowledge of either couple of the variables E_{ph} , E and θ it is possible to know the third one as a function of the known ones, this may be exploited e.g. to measure the spectrum of an unknown source which is too intense to be directly measured.

The efficiency or cross section of this process w.r.t. the various scattering angles shows a dependence from both the energy of the incoming radiation and (at lower energies) with the binding energy of the electrons in the sample: these features may be used to get information on the sample material as well.

The actual set-up for Compton scattering may vary following the application: in some cases the sample is also a detector so that the energy of the recoil electron is also recorded.

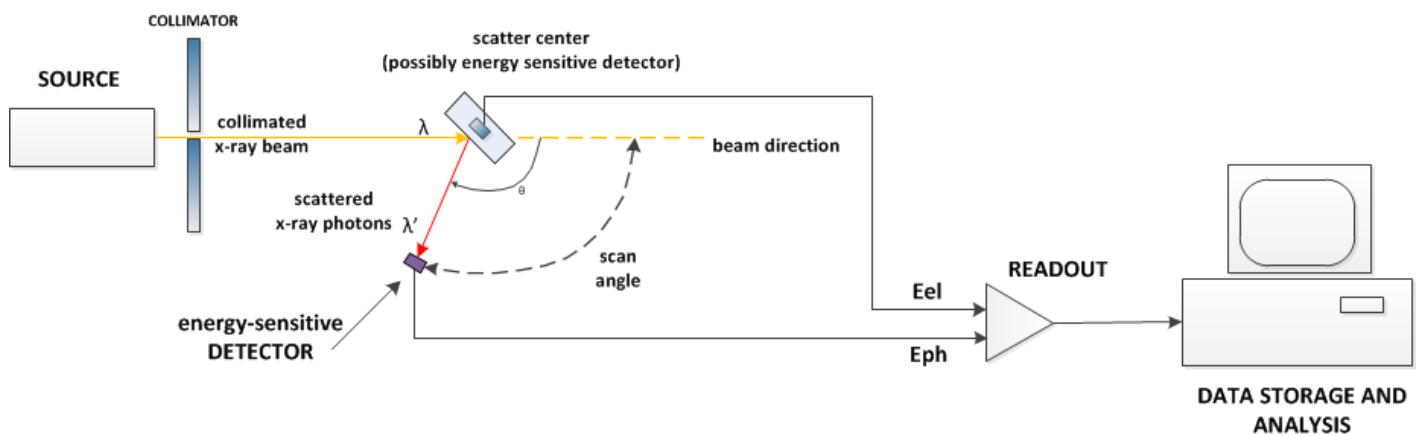


Fig 4.5.5.1: Sketch of a general set up for Compton measurements

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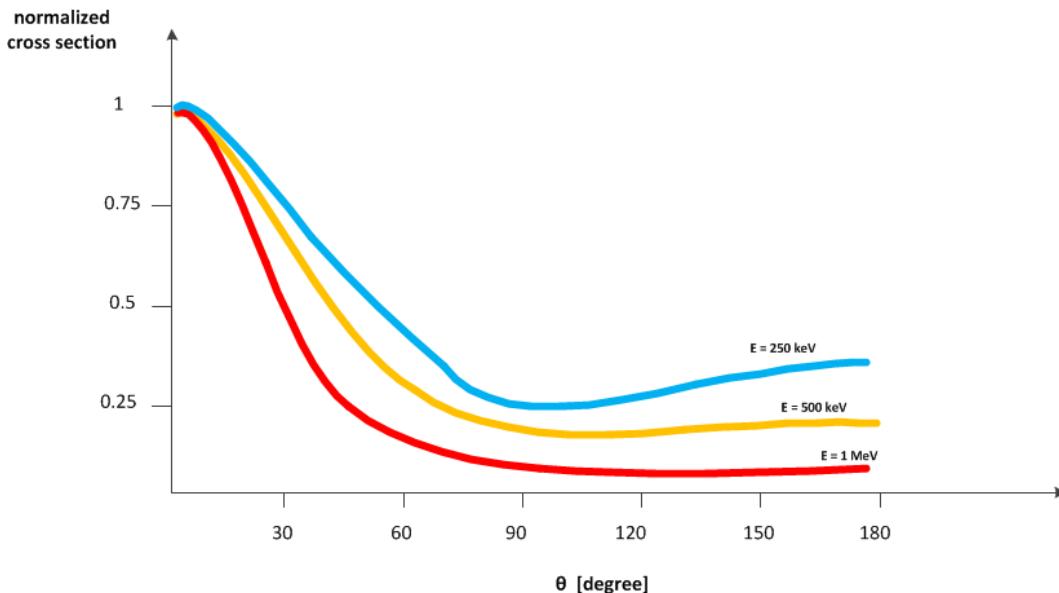


Fig 4.5.5.: Sketch of the Compton efficiency w.r.t. scattering angle (qualitative)

Main uses

spectral measurements of high flux, broad bandwidth X-ray sources

Discussion on potential improvement from AHEAD developments:

TES microcalorimeters optimized for gamma-rays or other detectors advances tackled in the frame of AHEAD WP9 may improve the performances of this technique, more likely in the field of basic research or material composition analysis.

4.5. PARTICLE INDUCED X-RAY EMISSION (PIXE) AND GAMMA-RAY EMISSION (PIGE)

This techniques has been around for a while [REF12]: it is based on the bombardment of the sample with high energy ions (typically protons in the 2-10 MeV range) so to ionize the inner shell.

Following reorganization of the atom characteristics X-Ray are emitted and can be analysed in a way similar to XRF.

The general set up include at least the following elements

- a collimated particle (protons) source to excite the atoms in the sample
- a x-ray detector
- an electronic chain (front-end + bias +back end for analysis)



The ion source is often a tandem accelerator which is well suited to the target energy range for protons.

One important feature is that it can be easily carried out in air (when using protons) providing that the x-ray detectors are close enough to minimize attenuation: this makes this technique well suited to the examination of e.g. art objects or natural specimen that could not easily sustain the high vacuum conditions normally needed for other techniques.

The beam-sample-detector area is usually flushed with gaseous helium which lowers the background due to interaction of the proton beam with air constituent like Ar which would emits x-ray lines in the energy range under study.

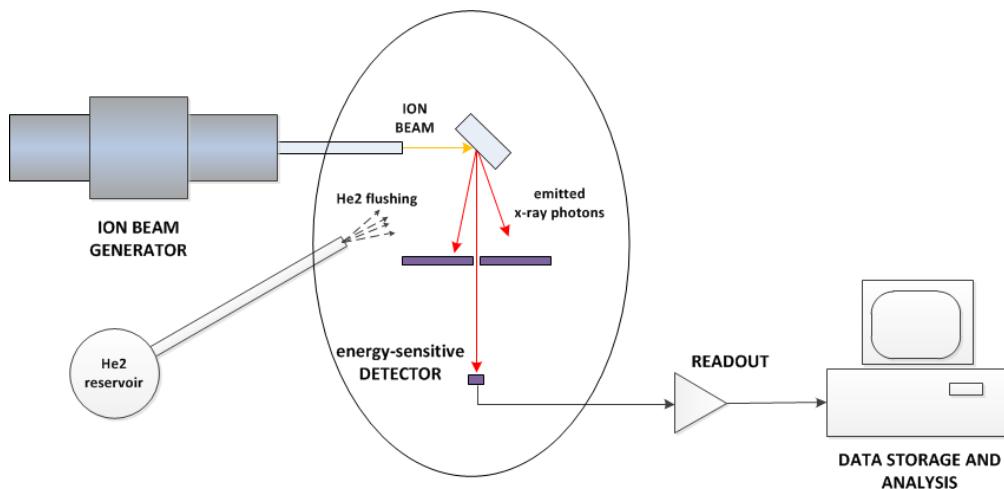


Fig 4.5.1: sketch of a typical PIXE set up

The end product is an energy spectrum of sample during de-excitation showing a set of lines and broadband intensity distribution reflecting the sample composition.

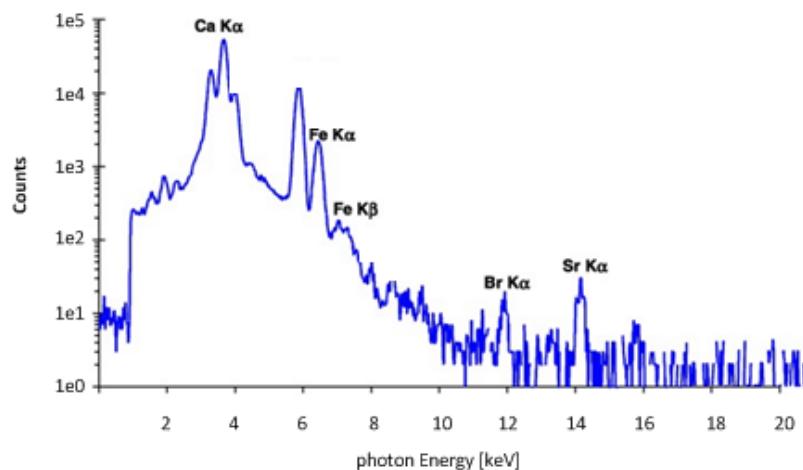


Fig 4.5.2: sketch of a typical PIXE spectrum

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A natural extension of this technique is the Proton Induced Gamma Ray Emission (PIGE) which makes use of higher energy protons (or other particles) to stimulate gamma emission from the sample nuclei.

Main uses

Elemental analysis

Trace element analysis (e.g. in air or soil for environmental studies)

Archeometry/Archaeology

Discussion on potential improvement from AHEAD developments:

TES microcalorimeters may improve the efficiency and resolution leading to a more accurate determination of the emitted photon energy.

Indeed this technique is one of the most promising due to its relatively easier set-up (modest energy proton accelerator and in-air operation) which could be improved using a detector derived from AHEAD development like an array of TES microcalorimeters for increased energy resolution and imaging capability.

Some studies in this field are already going on e.g. in Finland [REF10], [REF17] showing good results in terms of better element identification due to the reduction of interference between different lines in multi-element samples.

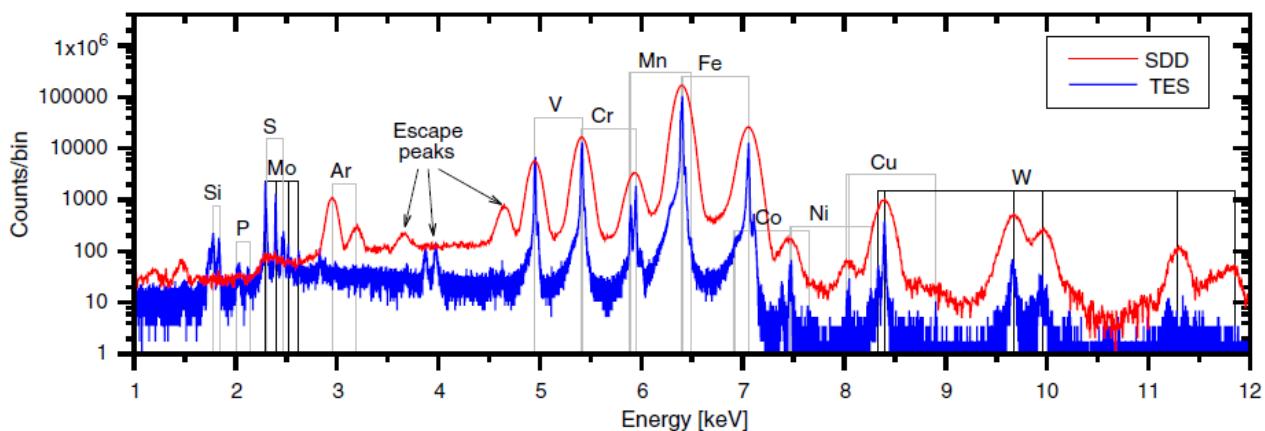


Fig 4.5.3 (excerpt from [REF17]): PIXE spectra of the same sample taken with a good “standard” detector (SDD, red line) and with a TES detector (blue line), note the much better peak discrimination obtained with the TES

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5. APPLICATIONS IDENTIFICATION PROCESS

After having identified, in the previous chapter, a set of experimental techniques which are related to the AHEAD technologies we here look at applications based on these techniques which are used in the scientific or industrial world.

We set a first distinction between applications for **space** which are the natural end-up of the AHEAD project, and application on **ground** that have possibly a much larger spectrum of end-users.

Ground applications usually benefit from a radiation source that can be tailored to the user needs in terms of energy, monochromaticity, coherence, intensity, collimation, and on-off switching speed.

The radiation sources panorama is in constant evolution, starting from radioisotopes, x-ray tubes, small scale accelerators and up to modern beamline facilities, each advancement also triggering a sprout of applications in various fields.

Space applications are mainly related to observation where the radiation source is natural and often very faint.

The AHEAD project is indeed focused on this type of applications and the resulting advancements are expected to be directly applicable to ATHENA or to other instrumentation (e.g. in the gamma-ray or IR or microwave domain) that can eventually benefit from the technologies developed in AHEAD.

The workplan for the survey has been to identify, both for ground and for space, an extensive list of disciplines and applications, in industry or research, which are presently using technologies studied in AHEAD.

The aim of this work is to collect for each application a picture of the state-of-art and to investigate if any of the AHEAD technological development could push the state-of-art further in terms of performances.

To do so we first collected and organized a large enough set of information based on specialized literature and on internal discussion with the AHEAD partners in the various research fields.

5.1. SURVEY RESULTS FOR GROUND APPLICATIONS

Albeit the mainstream of the AHEAD project is focused on spaceborn detectors for astrophysics we have identified some natural spin-off in terrestrial applications that could benefit from the high energy resolution of such detectors if their “civilian” implementation would be carried out down to an industrial product.

This holds e.g. for Material Science and Biology which could benefit from less invasive measurement and analysis as the introduction of the TES microcalorimeters will enhance the possibilities of doing non-destructive analysis of materials with low-dose tolerance like e.g. biological samples.



The following table gives a non exhaustive overview of the most important disciplines which make use of techniques that could benefit from AHEAD technologies

Biology/Life science	
toxic element build up in tissue/cells (by elemental/chemical analysis)	XES, PIXE
detection of blood contaminants (by elemental/chemical analysis)	XES
mass spectroscopy of biological molecules	TES as mass detectors
Medical/Veterinary	
Visual Diagnostic	
bones, tissues, inspection of implants	XRR, CT, PET
Functional studies, specific tissues (marked) visualization	PET
Physics/Chemistry and Material Science	
elemental characterization of sample (species, bonding, energy levels)	XES, XRF, XANES
chemical characterization of sample (molecules, energy levels)	XES, XRF, XANES
Environmental monitoring/survey	
air pollution (analysis of contaminants in airborne particles using air filters)	PIXE, XRF
soil pollution (analysis of contaminants in soil samples)	PIXE
water pollution (analysis of contaminants in marine/ocean sediments)	PIXE
Archaeology/archeometry	
composition of metals and alloys (historic studies on artifacts and finds)	XRF
characterization of paints, inks and pigments (dating and attribution of artworks)	PIXE, XANES
identification of gemstones (jewellery characterization)	XRD, XRF
detection of corrosion products (cultural heritage preservation)	XRR, XANES
internal analysis (non-destructive)	CT
authenticity	
Forensic	
crime scene traces analysis	XES, PIXE, XRF
crime scene material identification	
gunshot residues	XRF
Security	
luggage inspection	CT
weapons detection	XRR
dangerous/forbidden substances detection	XRF
Industrial QC	
Pharmaceutical	
Contaminant identification	XAS, XES
Drug identification by structure	XANES, XRD
Raw material composition	XES, XRF
Electronics	
RoHS compliance testing of raw materials and assembled parts	XRF
Wafer production	XRF
Detection of surface contamination	XRF
Detection of crystal defects	XRD
Consumer goods	
Detection of harmful substances in food/beverages	XRF
Detection of extraneous inclusions	XRR
Lead detection in toys	XRF
Petrochemical	
Fuel analysis, sulphur content	XRF

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In the following paragraphs the main families of ground applications are described and the possible use of technologies developed in AHEAD is discussed.

5.1.1. Applications in the field of Biology and Life Sciences

Microscopy has always been a fundamental technique in Biology and Life sciences as it is used to studies of life at different hierarchical levels like organisms, organs, tissues or cells.

In all these areas it is desirable to have high resolution imaging and this has led to the development of specialized techniques either in the optical domain or up to the electron microscope: all these methods usually requires a small dimension and/or transparent specimen and it is normally required to prepare “slices” samples to be observed.

The use of X-ray imaging allows seeing even through thick or large specimens and is becoming interesting in latest year due to both the development of innovative X-ray sources (e.g. Synchrotron beamlines) and innovative detectors which may extend the studies of biological systems down to the molecular and atomic scale.

At the present stage of development TES detectors are not well suited for this kind of applications due to the poor imaging resolution, if this could be overcome by some new readout approach, namely the micro-wave multiplexing, they could potentially be an advantage in terms of their potentially higher efficiency so allowing lower dose on the specimens.

5.1.2. Applications in the field of Medicine

Modern solid-state X-ray sensors are increasingly finding applications in medicine where they have already taken the place of former films in the up to date apparata. For most of these applications the final product is an image of a part of the patient's body showing details of internal tissues/organs that can be enhanced using proper contrast techniques to convey the required diagnostic information. These visual diagnostic applications usually requires imaging detectors with a high spatial resolution, commonly 2D systems or sometimes 1 D systems where the image is composed by scanning the subject. Up to now the requirement for energy resolution in these systems has been of less importance, apart from a rough energy discrimination capability to be exploited in the dual-energy radiographic technique to distinguish e.g. between soft tissues and bones.

In recent years it has been developed a new technique called sometimes “colour X-ray” which may be viewed as an refinement of the dual-energy method where the energy discrimination capability of detectors used in radiography or CT has become important to allow accurate identification of different tissues (e.g. muscles, blood vessel, bones, cartilage and so for) so providing a much more clear insight into the patient body.

The detectors used for these Colour X-ray systems are presently CMOS detectors or CCD working in single-photon counting mode with an inherent energy resolution in the order of 100-200 eV while allowing spatial resolution down to few tens of µm.

TES detectors may improve these systems by virtue of their superior energy resolution but to really compete they will have to increase the achievable spatial resolution.

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5.1.3. Applications in the field of research for Material Sciences, Physics, Chemistry

X-Ray are customary used in these research fields starting from the very discovery, X-ray spectrometry has been used to identify the elemental composition of materials and X-ray diffractometry has been used to study crystal spatial arrangement.

In recent time the emergence of high resolution X-ray spectrometry and of new intense and tunable sources is leading to more sophisticated capabilities including the determination of the chemical state of atomic species within compounds so allowing a complete physical-chemical characterization of the sample.

One important development line is the materials analysis within Scanning Electron Microscopes (SEM) which is used in many laboratories for elemental characterization by XRF produced by the electron beam.

TES detector could be beneficial in this “microbeam” technique as they can provide at the same time a better energy resolution w.r.t. the commonly used SDD detectors and a greater efficiency in particular at the higher energies. Imaging capability in this kind of application would be desirable but not of fundamental importance as the spatial reconstruction of the elements can be obtained by scanning the microbeam across the sample.

Despite the foreseeable technical challenge, placing a TES detector in close proximity of the sample within a SEM chamber would be highly attractive in particular if the TES energy resolution was in the order of few eV as it would allow the chemical analysis of the sample.

Another promising development line is the material analysis at large scale facilities like synchrotrons or XFEL: these facilities allow for high intensity beams with tunable energy and often also allows for very short pulse stimulation (< 1 ps can be reached with XFEL).

The high intensity of these beams cannot directly be exploited by TES detectors being these intrinsically low-speed (thermal) devices but on the other hand it can be used to enhance the photon yield of low-efficiency techniques like e.g. XRS.

Also in this case the superior energy resolution of TES detectors can be used to perform detailed analysis of the chemical state of the atoms in the sample.

Another promising development which is in a somehow intermediate rank between SEM and beamline facilities is the use of TES detectors in PIXE facilities which have some advantages w.r.t. SEM in the fact that the protons which are commonly used produces lower bremsstrahlung background than electrons so allowing the detection of small concentrations materials.

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5.1.4. Applications in the field of Environmental Science

There is an increasing worldwide awareness of the importance of controlling and protect the environment, also enforced by stringent laws and regulation at least in the more developed countries. Pollutants from agricultural or industrial processes can contaminate soil, water and air with toxic substances that can affect many products for human use, in particular food and beverages.

To detect and control these pollutants it is common to analyse environmental samples of soil/air/water searching for dangerous elements, in particular heavy metals like lead, cadmium and mercury which have a well-known toxicity. These contaminants should be measured in the parts-per-million concentration range so that a high sensitivity is required and X-Ray techniques are particularly efficient in this detection.

There are many techniques for elemental analysis, the most important being EDXRF (used in the field) and PIXE for extensive studies in laboratory.

In particular PIXE is used to identify fine particle pollution in atmospheric air passing the air at a constant flux through special Teflon filters which retain the particulate down to a specified size.

This air particulate pollution can be either by human activities (anthropogenic) like industry, house heating or vehicles or from natural causes like volcanic eruption, forest fires, windblown fine dust or saltwater spray above the seas.

The filters are used as target for a proton beam and the resulting X-ray emission is analysed to identify the particles elemental composition. PIXE can typically provide useful data for elements from Al to U and can also perform trace element analysis i.e. detecting very low concentration against an embedding matrix.

Using PIXE it is possible to create and maintain a large database of the elemental concentrations based on daily, or even hourly, samples collected by automatic air sampling devices using very small quantities of particulate mass. The identification and quantitative characterisation of the pollution sources are useful for the environmental protection agencies and at legislative level.

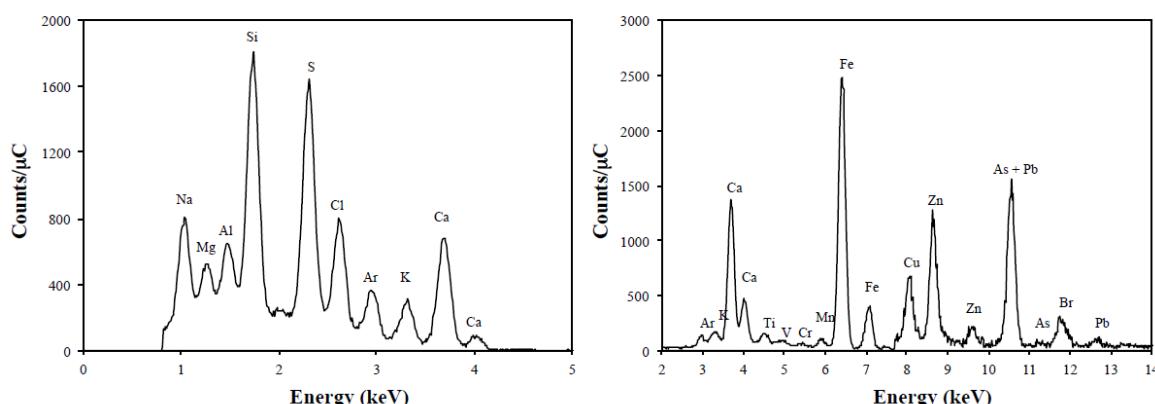


Fig. 5.1.4.1: example of PIXE spectra (PM₁₀ sample, industrial area)

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The PIXE technique is routinely used to study air particulate pollutants in critical areas of the world using semi automatic analysis which allow to process daily of hourly samples with the following typical yields:

Daily samples: 1 filter/day, 1 min/filter, 1 year of data taking = 8 hrs analysis

Hourly samples: 1 filter/week with 7x24 sectors, 1 min/sector, 1 week data = 4 hrs analysis.

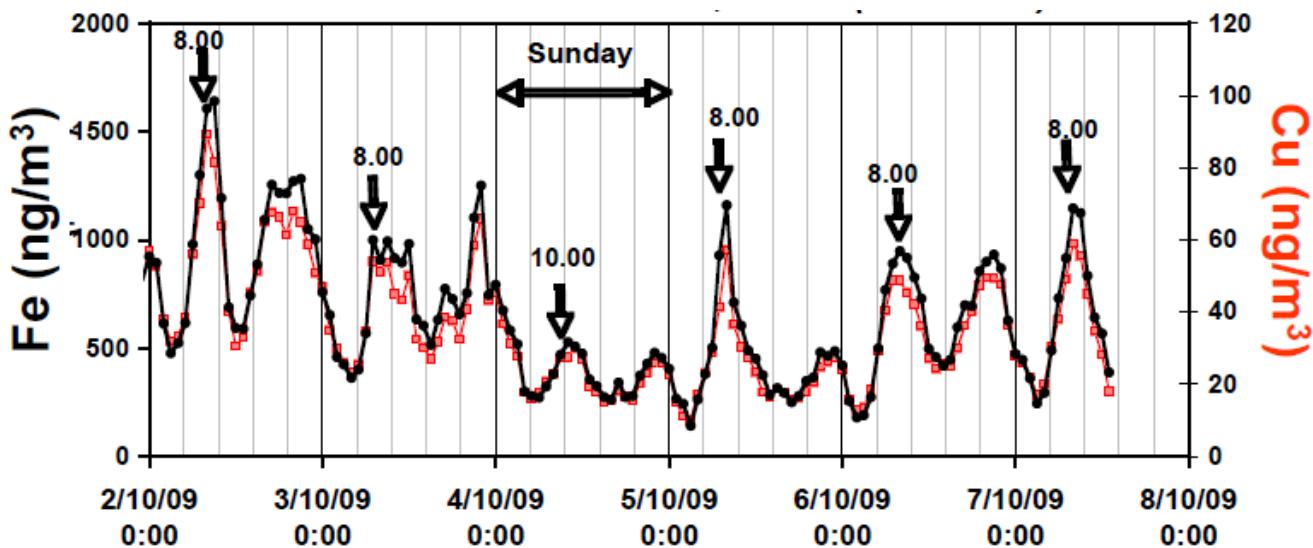


Fig. 5.1.4.2: example PIXE analysis of hourly sampling air pollution in one week in Florence.

The figure above (credit LABEC) shows the concentration of Fe and Cu having peaks in the morning rush hours and a lower concentration during the weekend.

The detectors used for most of existent PIXE facilities are presently SDD detectors due to their availability and ease of use, anyhow from the survey on their use in air pollution analysis we see that the use of TES detectors in PIXE could provide an important improvement in the capabilities of these systems due to both the better energy resolution and the larger efficiency at higher energies.

5.1.5. Applications in the field of Cultural Heritage, Archaeology, Arts

Modern techniques of material analysis are of great importance in the field of cultural heritage and many X-ray techniques are currently used e.g. to assess the origin of the artefacts through the precise identification of the used materials like pigments (e.g. detecting cadmium and chrome based pigments on paintings), metal alloys (e.g. detecting traces of silver in bronze) or determining the gold karat level in a piece of jewellery.

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In particular PIXE is relevant in the field of material identification through the analysis of the major constituting elements, to material provenience through trace analysis and to manufacturing technology by micro-PIXE (i.e. spatial resolved PIXE, scanning the subject with micro beams).

An important feature of PIXE is that the object to be studied can be maintained in standard atmosphere pressure so allowing a true non-destructive analysis of delicate samples.



Fig. 5.1.3.1: example of PIXE analysis of an ancient manuscript (LABEC)

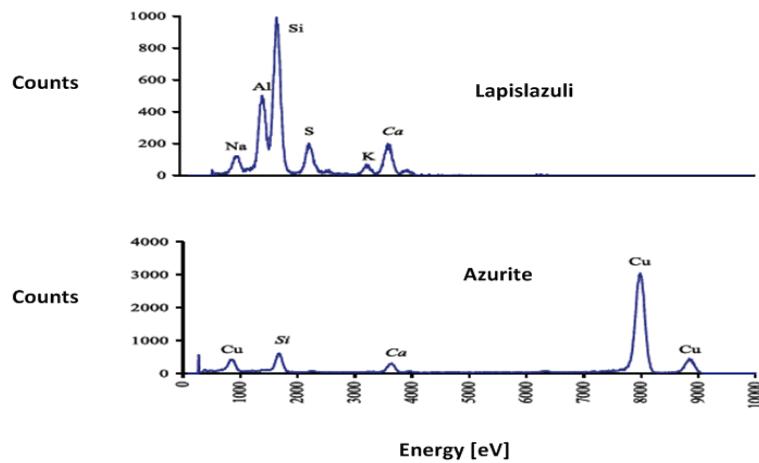


Fig. 5.1.3.2: example of PIXE spectra from two different blue pigments (LABEC)

Also in this fields, as said in the case of Environmental Science, the use of TES detectors in PIXE could provide an important improvement in the capabilities of these systems due to both the better energy resolution and the larger efficiency at higher energies.

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5.1.6. Applications in the field of Forensic

Forensic practice often have to rely on X-ray techniques to analyse a crime scene in search of clues of what actually happened, e.g. for qualitative analysis of explosion by-products, analysis of bullet residuals, trace of metals in human tissues or other material analysis e.g. for detection of toxic substances.

The main use of X-ray techniques in this context is for elemental analysis of crime scene samples of various nature, a common need being that of preserve the sample integrity so that it could later be used as “evidence” in a trial so that only non-destructive techniques can be used.

The nature of samples may be very undefined and composition unknown a priori so that the technique to be used for analysis should be targeted to as many elements as possible.

Also in this case the most important technique is EDXRF but PIXE could also be envisaged for special cases involving delicate samples where the adoption of TES detectors could mate it with particular investigation requirement in terms of trace elemental or even compositional analysis. At present it is not known if PIXE has ever been used in this field, nevertheless it is one potential, albeit niche, application to be taken in account.

5.1.7. Applications in the field of Security

Security is one of the largest field of application for X-ray technology, from basic radiographic system used to detect metal weapons in luggage or on body to more advanced system capable of distinguish the different materials used as explosive compounds.

5.1.8. Applications in the Industry for QC and NDT

Quality Control and Non Destructive Testing are important elements of the Quality Assurance process in the modern production lines including both agricultural and industrial fields.

There is an increasing consciousness in the importance of “Quality” to be delivered to the customers and this requires an intense QC based both on the installation of proper inspection equipment directly on the product lines and on sample-based laboratory inspections.

The QC equipment should be as much as possible automatic, easy to install and maintain e.g. on the conveyors carrying raw materials and intermediate/final products.

The outputs of these QC equipments should be made quickly available at the line inspector to allow immediate reaction in case of non-conformance.

X-ray systems are often used in industrial QC and NDT activities and there is a large number of off-the shelf instruments available on the market based on XES or XAS or XRD depending on the particular application.

All these instruments can be classified at a first level in the two categories of:



- a) Imaging systems
- b) Non-imaging systems

The present Imaging systems, based on CCD or CMOS detectors, are at a level of number of pixel / spatial resolution which can not likely be tackled by TES systems while the asset of TES microcalorimeters is more in the non-imaging system where the energy resolution is of utmost importance.

These non-imaging applications include e.g. on-line inspection of drugs components in the pharmaceutical industry, on-line inspection of contamination in the food & beverage industry or detection of contaminants in the electronic production lines (wafer-level). Some of these systems may take advantage by the increased energy resolution and efficiency made available by TES microcalorimeters to improve their performances in terms of element identification against a disturbing background.

5.2. SURVEY RESULTS FOR SPACE APPLICATIONS

In the last 30 years there has been a continuous development in the field of cryogenic detectors and cooling technologies in terms of reliability that have making possible to use these technologies on-board spacecrafts.

At the same time the progress in cryogenic sensors and "cold" electronics has paved the way for new scientific instruments.

Many space missions have already been flown including some cryogenic equipment, in particular in the field of astrophysics observing at wavelengths not achievable by ground observatories due to the atmospheric absorption. Examples of such pioneer missions are the ESA InfraRed Astronomical Satellite (IRAS) and Infrared Space Observatory (ISO), the Japanese ASTRO-E targeted to X-ray astronomy and the USA Cosmic Microwave Background Explorer (COBE). More recently the ESA PLANCK observatory has successfully made use of deep cryogenic technology for the most complete CMB anisotropy mapping to date.

For the near future there are both in Europe and USA and Japan many studies relating to space missions based on cryogenic technology, the flagship being the ESA Cosmic Vision large mission ATHENA devoted to High Energy astrophysics.

The interest in cryogenic detectors over other more common sensors is based on their higher sensitivity when used as bolometers and high energy resolution when used as calorimeters.

This ever growing interest for the space scientists has led to an increasing demand of cryogenic systems in space affecting the system-level architectural design of the related spacecrafts in terms of power generation and distribution, thermal design to obtain and maintain the desired

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temperature, temperature stability (often requiring L2 orbits) and in general posing stringent requirement on the mission design.

Cryogenic detectors and related readout technology are proposed for non-dispersive spectroscopy e.g. in X-ray photon-counting applications. In particular the TES have very good performances in X-Ray with higher detection efficiency, photon counting and intrinsic spectroscopic capability w.r.t. more common CCD or CMOS detectors albeit more difficult to overcome them in terms of spatial resolution.

The use of TES detectors (and related technologies) in space is indeed at the focus of the AHEAD project. The sensitivity and energy resolution achievable with this kind of detectors is appealing in particular for scientific instruments while the intrinsic costs associated to space instrumentation are such that the burden of cryogenic technology is of relative less weight than for ground instruments.

On the other hand the complication of cryo-cooling down to sub-K level in space is a formidable technical challenge due to the low-efficiency of available cooler system together with the limited amount of power available on board spacecrafts.

For applications requiring the measurement of radiation flux rather than photon counting, as is the case for IR astronomy or mm-wave astronomy, the TES detector used as bolometers are also a resource and the on-going development have already obtained NEP (Noise Equivalent Power) down to 1e-19 Watt/sqrt(Hertz).

Also the SQUID devices that are commonly used to readout the TES detectors are of interest in their own for instruments for fundamental physics or planetary science as e.g. in experiments to verify the Equivalence Principle or in precision gravity measurements.

The application of TES detectors in space is presently envisaged in different scientific fields and different observables taking advantage of the possibility to “tune” the TES detector to the required target. The following table resume some space applications which are presently under study or development.

Scientific domain	Target observable	TES use	Remarks
High Energy astrophysics	x-ray typ 1-20 keV	microcalorimeter	reference mission: ATHENA (ESA)
Cosmology	CMB millimeter waves typ 10-500 GHz	bolometer	reference mission LiteBird (JAXA)
IR astronomy	FIR typ 30-200 μ	bolometer	reference mission (SPICA) JAXA-ESA
Solar physics	x-ray typ 1-20 keV	microcalorimeter	proposal phase in USA

From the survey carried out so far it is apparent that cryogenic detectors are under technological study in the many important space organizations and it is very likely that at least a

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few science missions based on TES detectors will be implemented in the next decade. Other emerging detector technologies (e.g. Kinetic Inductance Detectors) are also under development but still at less developed stage w.r.t. the TES.

Ancillary subsystems like cryo-coolers and specialised readout using SQUIDs are also being developed so opening the way for the use in space of scientific instruments based on cryogenic technologies of different types.

5.3. IDENTIFICATION OF THE TARGETS FOR FOLLOW-ON ACTIVITIES

This survey has given us a panorama of the quite large field of X-ray techniques and has allowed to select a few promising applications for investigation in the second phase of the project.

These applications have been examined in terms of their potential improvement in performance using TES microcalorimeters as X-ray detectors, after the first screening the most promising ones resulted the following (as discussed in the AMT meeting #2, March 2017):

Ground:

- | | |
|-------------------------------------------|-------------------------------------------------------|
| Composition analysis by PIXE | (for environmental studies and for archaeology / art) |
| Probe at XFEL/Synchrotron facility | (for basic research on material science) |
| Composition analysis by XRF | (general analytical use) |
| Composition analysis by PIGE | (for land mines detection) |
| Medical imaging CT/PET | (for diagnostic) |

Space:

- Space instruments for x-ray spectroscopy** (mainstream focus of AHEAD)
- Space instruments for observation in the IR and in the microwave bands**
- Space instruments for sun observation (corona)

The highlighted applications have been selected for further investigation and those related to terrestrial use are briefly described herebelow, while the space applications are deeply dealt with in the reporting from the other relevant WPs.

The introduction of AHEAD technologies in medical/veterinary diagnostics is still a field to explore: albeit most of these applications are based on photon-counting rather than spectroscopic detectors the TES microcalorimeters could potentially provide advantages in terms of increased counting efficiency and better background discrimination like e.g. in Computerized Tomography systems for visual diagnostic.

Some applications that have been among those identified as promising in a preliminary selection have been later recognized as not viable due to technology limits which are beyond the horizon of AHEAD. This was the case for landmines localization which requires high energy detectors (in the MeV range) which do not match well with the TES microcalorimeters for x-ray

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which are the main focus of AHEAD. A survey of previous works on this subject has been carried out without finding a supporting case, for this reasons this application has been put at lower priority in the discussion held at the AMT meeting #1 in October 2016.

5.3.1. Composition analysis using PIXE

As described in chapter 4.5 the Particle Induced X-Ray Emission (PIXE) spectroscopy is based on the bombardment of the sample with high energy ions (typically protons in the 2-10 MeV range) so to ionize the inner shell of the sample atomic species.

This technique has been selected as one of the most promising for possible follow-up of the technologies developed for AHEAD.

We studied the possible advantages of the introduction of TES detector chain in this kind of set-up, typically in the form of a compact measuring head with few detector channels (possibly with different absorbers to allow optimization at various energy ranges).



Fig . 5.3.1.1 a painting under examination at a PIXE facility
(credit INFN, LABEC laboratory)

To investigate the practical application of the AHEAD technologies we started a collaboration with one existent facility in Florence (INFN LABEC) for a co-engineering study with focus on the accommodation constraints for the detector head and for the identification of the kind of measurement/analysis which could make most from the adoption of TES.

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The LABEC laboratory could eventually provide the facility to test a prototype of such spectrometer in a real operative environment for applications including:

- Air pollutant identification: the samples may be fabric-like filters which have been fluxed for a given volume-time with the air in the place to be monitored so collecting pollutants according to the filter composition. The filters are collected along a given time sequence (e.g. one per hour for a whole week) and stored in tight container. At the moment of analysis each filter is placed in front of the beam and the fluorescent spectrum is recorded.
- Fine arts: the sample, e.g. a portion of a painting is directly exposed to the beam while the fluorescence x-ray spectrum is recorded to identify the elements composing the pigments used by the artist. The improved resolution would allow disambiguation of overlapping lines e.g. for S, Pb, Ti, Ba allowing better identification of such pigments.
- Archaeology: also in this case the beam is fired on specific portion of an item to identify its composing elements. This is useful e.g. to identify alloys composition in metallic artworks
- Geology: tracing rare earth elements in mineral samples
- Gemmology: characterize diamond purity to ppb level
- Forensics: many application e.g. to identify fake pharmacological products or to analyse specimen from a crime scene to detect the presence of valuable hints.

The technical details of the probe under study are given elsewhere (D10.6), from discussions with LABEC we have identified two target applications for the probe as follows:

- a) Low-Z targets: these are characterized by emission lines mostly in the < 6 keV range and an high cross-section so leading to a high yield of X-ray photons to be analyzed. These conditions allows the use of set-up geometries with a small solid angle which is suited to the low area detectors which are easier to be implemented with TES. Moreover the characteristic X-ray lines in this range are often close-spaced and the high-energy resolution achievable with TES could provide a real advantage in disentangling. The typical PIXE application in this range are the analysis of aerosol samples and cultural heritage specimens.
- b) High-Z targets: in this case the cross section is lower and a larger solid angle is required in the set-up geometry to collect a significant number of photons, also the statistics of counts is prevailing over the noise so that TES are generally not a great advantage unless they are designed for significant higher efficiency w.r.t. to standard silicon detectors.

For applications of type a) a solid angle in the order of 2 msr is acceptable while for applications of type b) it should be targeted a solid angle at least two order of magnitude larger.

The following figure shows a sketch of a typical set-up geometry and the solid angle definition.



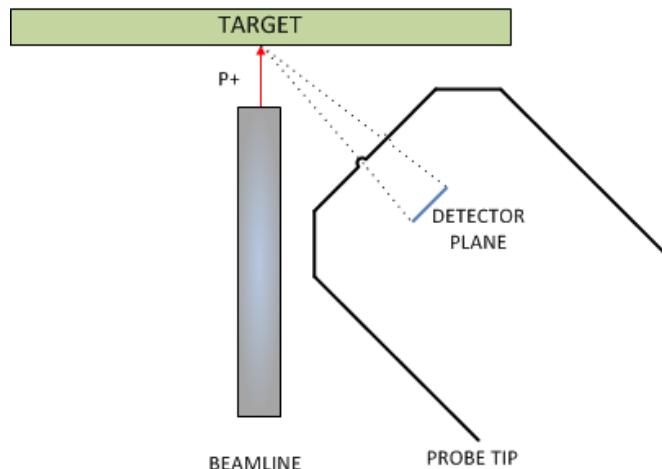


Fig . 5.3.1.2 sketch of the probe-target geometry for use in the LABEC facility

Another distinction that can be made for PIXE application is that between “majority elements” and “trace elements” applications: the first case is easier to deal with while for traces there is the problem of the background elements masking the useful signal: in this second case the better resolution of TES detector could be an advantage in making the trace lines “emerge” above the background spectrum.

5.3.2. Probing Station at an XFEL or Synchrotron radiation facility:

Synchrotron radiation facilities with x-ray generation capability are routinely used for material science research and have allowed in the last tens of years major advancement in this field.

X-ray free-electron lasers (XFELs) are the ultimate x-ray source for the most advanced scientific investigations which rely on x-ray to probe matter. XFELs generate coherent, ultra-brilliant, tunable x-ray laser pulses with femtosecond pulse durations that allow to resolve the structure and dynamics of matter on the atomic scale.

Their performances in terms of monochromaticity and brilliance overcome that of synchrotrons by order of magnitudes making them the ideal source for most of the techniques identified in the previous chapter.

Like synchrotrons, XFELs are quite large facilities that are currently being developed in the most advanced countries in the world to enable progress in many scientific fields from chemistry to physics and materials science and also biology and life science.

The introduction of a spectrometer based on TES microcalorimeters at such large facilities is likely easier than in small scale laboratories in terms of resource balance (cost, complexity and space occupation) and will allow better performances experiments based on inelastic x-ray scattering for material science research at atomic level.

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From the above considerations we see that another possible contribute of the technologies developed in the AHEAD project may be the development of a research-grade TES microcalorimeter to be used at such large facilities like XFELs and Synchrotrons.

In our view a compact TES detector measuring head plus the related readout chain could become a sort of “bench” instrument to be used for many pure and applied research investigations in the field of materials science.

5.3.3. Composition analysis by XRF:

As described in chapter 4.2.2 this technique finds application in the field of chemical research, in particular for the identification of bonding states of the atoms in the particular substance being analyzed.

It is a much versatile technique for non-destructive elements identification from Sodium (Na) to Uranium (U) where the sample may be powder, liquid or solid.

Used also in hazardous substance detection and alloy composition identification.

The introduction of TES microcalorimeters with resolution at eV level may improve the spectroscopic performances of these systems. Moreover the TES microcalorimeters may be designed to be more efficient than usual Si detectors in the harder x-ray range.

These features can be very useful in experiments with low intensity source beams, either due to source limitations or when analysing dose-sensitive samples.

In recent times TES microcalorimeters arrays are being used in XRF applications associated with electron microscopes which can “naturally” provide the required excitation.

From the above considerations we deem that there possibly is an opportunity for the development of a compact laboratory-grade TES microcalorimeter spectrometer for general analytical use, possibly coupled with an electron microscope.

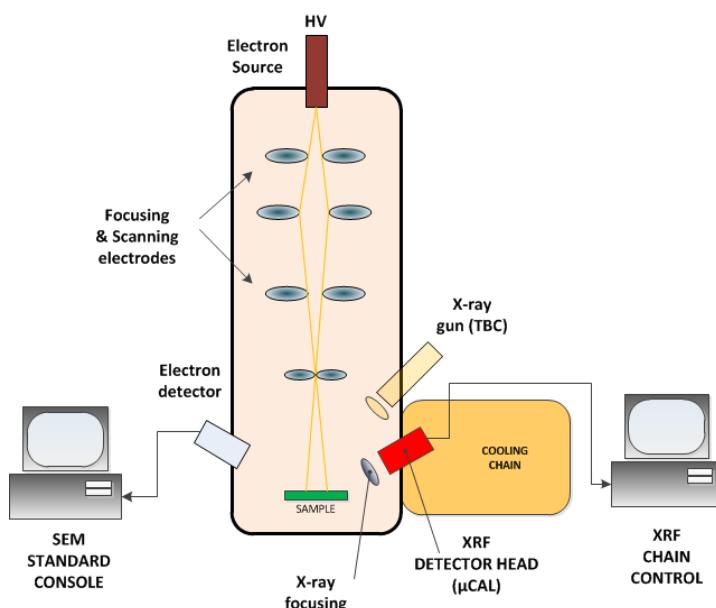


Fig . 5.3.3.1 sketch of a TES chain coupled to a scanning electron microscope



We investigated this possibility taking it in account in the frame of the development of a compact TES detector head for laboratory use.

5.4. SELECTED FOLLOW-ON ACTIVITIES

The three “ground” application that have been selected as suitable for a follow-on may be characterised by having a set of common components listed herebelow:

- X-ray Optics, as a minimum a collimator and a filter to block IR radiation
- A compact cold head (detectors, squids, flanges for cooling)
- A cooled-vessel matched with the above cold head, capable of high vacuum and including a cooling chain down to sub-K level.
- A specialised readout electronics (SQUID front end + back end)

The selected “ground” applications presented in the previous chapter are all suited for single or few-pixel detectors not strictly requiring multiplexing.

The system requirements study carried out in the frame of AHEAD has suggested a step by step approach in the technology transfer roadmap.

For this reason the follow-on system engineering study has been aimed to a subset of the reference detector system which is deemed to have an early chance for implementation and has been devoted to a “GROUND” system while maintaining “SPACE” compliance in account whenever possible.

The system engineering study has then been focused on two building blocks:

- a) a compact/interchangeable DETECTOR HEAD module with single or few detector channels (detectors +magnetic shield +optical filters +cold-FEE +cryo-harness)
- b) a suited readout electronics (WFEE, WBEE)

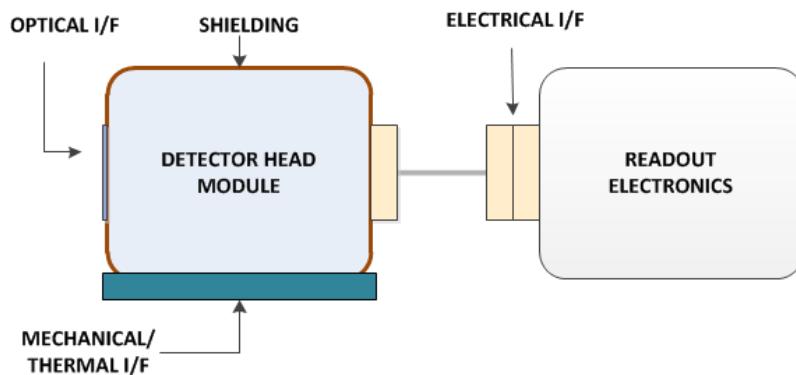


Fig . 5.4.1 building block to focus on in the system engineering study

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Considering all other subsystems elements to be taken “off the shelf”.

This engineering study has been carried out up to the definition of a reference design for a probe specifically designed for PIXE applications and based on a small array of TES pixels in a not-multiplexed readout scheme.

The system described in the engineering study could be proposed for actual implementation in a future development phase.

Further on a more sophisticated system has also been envisaged in the final phase of the AHEAD project which takes the opportunity from the advancement in the multiplexed readout techniques that have recently taken place and foresee the implementation in the PIXE probe of a small size array of pixels (15 to 36, TBD) readout using FDM multiplexing with an architecture derived from what foreseen for the ATHENA XIFU spectrometer.

This later system albeit being more complex to be implemented and tested would give the advantage to exploit the ultimate TES performances in terms of energy resolution while at the same time paving the way for the production of larger arrays with true imaging capabilities.

The end-to-end implementation of a prototype of the multiplexed array system is currently being proposed for the next “AHEAD-2” project following the H2020 call due in Q1 2019.

END OF DOCUMENT

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