Laboratori Nazionali di Frascati

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NEW SCATTERING CAMERA FOR ANISOTROPIC ULTRA-LOW ANGLE SCATTERING EXPERIMENTS USING A THREE-DIMENSIONAL IMAGING GAS DETECTOR OPERATING WITH ESRF X-RAY SOURCES

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INTRODUCTION

The availability of new very high brilliance X-ray synchrotron radiation sources in the near future could allow to extract a lot more informations from a low-angle scattering experiment. However this can only be achieved when the performances of the low-angle scattering cameras are optimised for an unambiguous identification of reflections arising from the sample and a quantitative analysis of the shape and the time evolution of scattered radiation intensity. Thus for anisotropic scattering measurements (e.g. for the investigation of collagen fibrillar organization or the determination of faceted metal void orientations, etc.) scattering cameras with symmetrically circular beam and two-dimensional detectors are required. A scattering camera with these features requires pin-hole collimated beam and a detector with high spatial resolution on both coordinates together with a high time resolution and a high count-rate capability.

The performances of the most used two-dimensional detector (TV systems; multiwire proportional chamber: MWPC; charge-coupled devices: CCD; multi-channel plates: MCP) are far from these ideal requirements.

The present commercial matrices of CCD have low dynamic range, are noisy and suffer from radiation damage. The MCP devices have low efficiency, low count-rate capability and their response is "not constant" for different incidence angle of the photons. TV systems can be useful only for high scattered intensity, because their response is noisy and not uniform on the whole detecting area. Multiwire proportional chambers could be potentially versatile, but at present their response is not uniform on the whole of the detecting area due to the wire structure. Additionally their spatial resolution is not high.

The three-dimensional imaging drift chamber, a proportional gas detector in the class of multiwire chambers, developed by us at Frascati, has better performances especially in the spatial resolution and in the uniform response on the whole area(1).

Therefore we propose to build for ESRF an X-ray scattering camera with pin-hole collimation and our drift chamber gas detector which will be suitable for anisotropic scattering experiments in the range of ultra-small and medium-angle scattering. This apparatus can be considered a natural development with better performances of that operating at Adone storage ring.

SCATTERING CAMERA

The scattering camera can be designed to operate with ESRF - 30 pole wiggler source and with standard asymmetric cut crystal monochromator, but can be used with each other ESRF source.

As shown in Fig. 1, this instrument consists essentially of a pinhole collimation system, a specimen chamber and a two-dimensional drift chamber detector. The collimation system and the sample holder chamber are assembled in a single vacuum chamber, while the detector carries a vacuum telescopic tube for collecting scattered radiation. The whole camera system

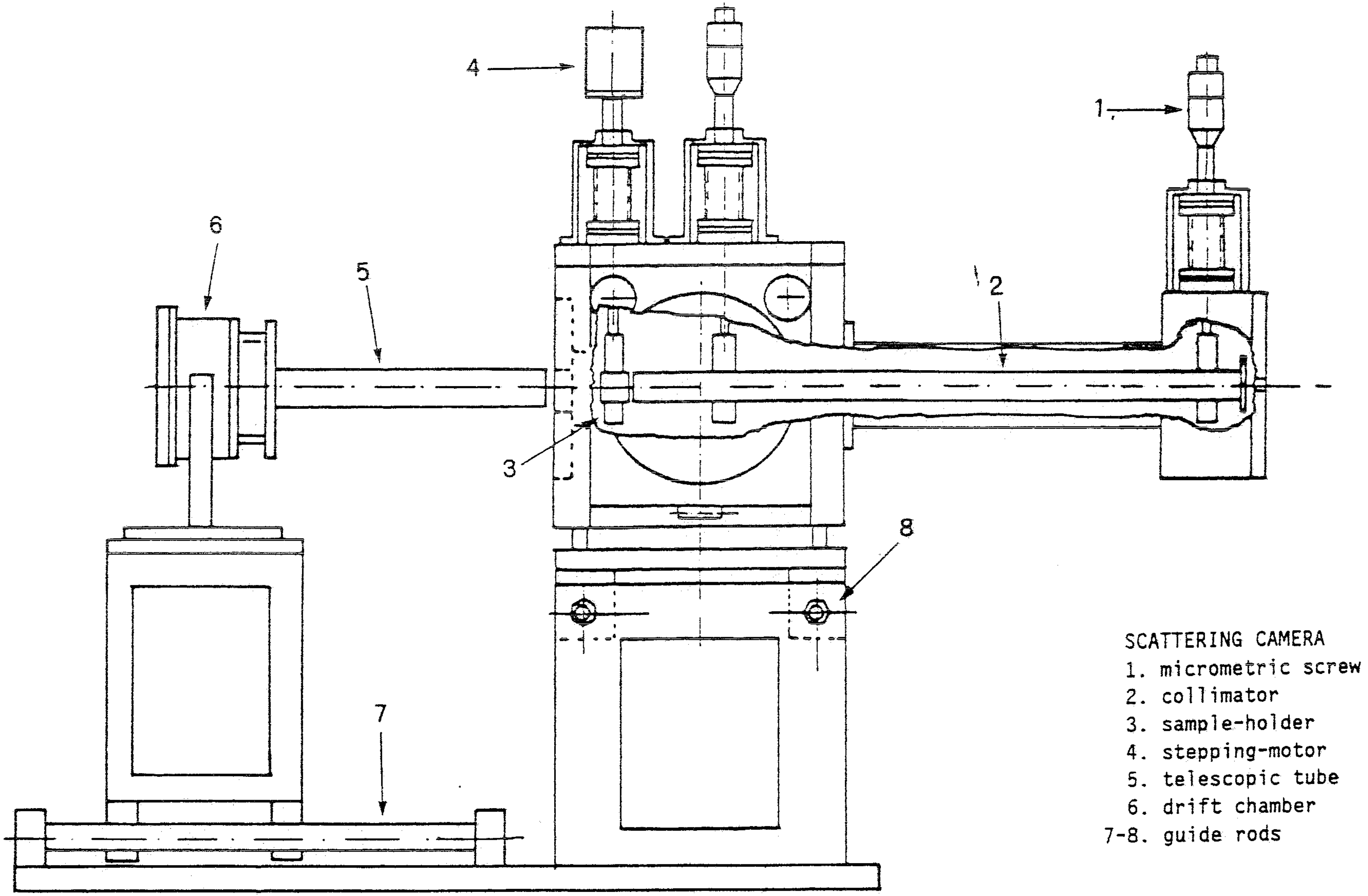

FIG. 1: General view of the Scattering Camera.

at pressure of 10^{-3} bar is mounted on smooth guide rods in order to be able to move on the horizontal plane perpendicularly to the beam-line. This movement makes it possible to draw away the specimen chamber from the beam-line, for the purpose of mounting the sample inside. At the same time it is possible to align it with the collimator using adequate support optical instrumentations (as required by very small sample).

The basic geometry of scattering camera is show in Fig. 2. A single pin-hole F_1 is sufficient to collimate the beam on the sample because the source is very small. For cutting off the parasitic radiation genered from the edges of the pin-hole F_1, a second pin-hole F_2 has to be located at some distance d_1 from F_1(3).

For anisotropic scattering experiments a small light spot hitting the sample is necessary to irradiate homogeneously the sample with a small homogeneous beam. An estimation of divergence at the collimated beam can be made by using optical values of the diameter of $F_1 = 0.5$ mm, $F_2 = 0.7$ mm, $\ell = 30{,}000$ mm, $d = 2{,}000$ mm and $\sigma_x = 0.062$ mm, $\sigma_y = 0.040$ mm for source size. This is $2\alpha = 1.8\times10^{-5}$ rad. The acceptance angle of the system $F_1 - F_2$ is $2\beta = 6\times10^{-4}$ rad. Because $2\alpha < 2\beta$, X-ray beam impinging on F_1 is always confined within the angle 2β. The adjustement of the hollow cilinder is carried out by four micrometic screws mounted on the vacuum bellows. The sample holder is also guided by other two external micrometric screws for aligment at the sample with the beam.

For the geometrical dimension used the value of the scattering vector $h = \frac{2\pi \times 2\theta}{\lambda}$ is 2.5×10^{-3} Å^{-1} and the maximum detectable diameter of scattring unit $D = \frac{2\theta}{\lambda}$ is 2,500 Å. For a beam size of 0.2 mm^2 the incident flux is 0.4×10^{13} photons/sec at $\lambda = 1.54$ Å.

In this chamber solid sample and cells for liquid samples, exposed also to external hydrodynamic field, must be placed at short distance of F_2 and near to exit window of system to allow collection of scattered radiation also at medium angles.

Detector is also mounted on a system of smooth guide rods to move it parallely to beam-line and to take it near the sample in a range of

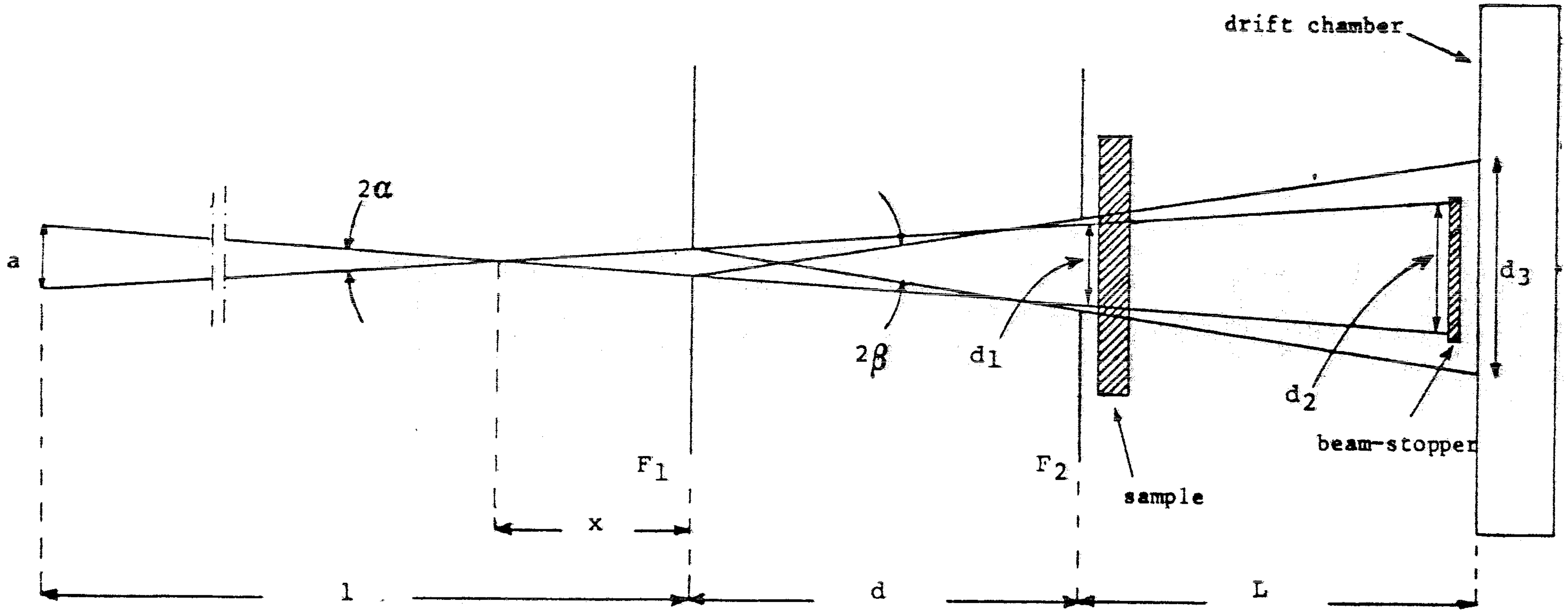

FIG. 2 : Basic scheme of the Scattering Camera: a = source size; l = 30,000 mm; d = 2,000 mm; L = variable distance in the range 40-2,000 mm.

40-2,000 mm.

In that case the solid angle of detection is increased and it is possible to record medium angle scattering patterns (this is useful for the investigation of morphological modifications in bone collagen calcified fibrils).

Vacuum telescopic tubes for collecting scattered radiation at different distance are linked to the detector. A beam stopper (movable by micrometer screw) is placed at a few mm. from the entry window of the detector.

DETECTOR

The scheme of the two-dimensional drift chamber gas detector is shown in Fig. 3. The chamber has a drift region between grid and field electrode.

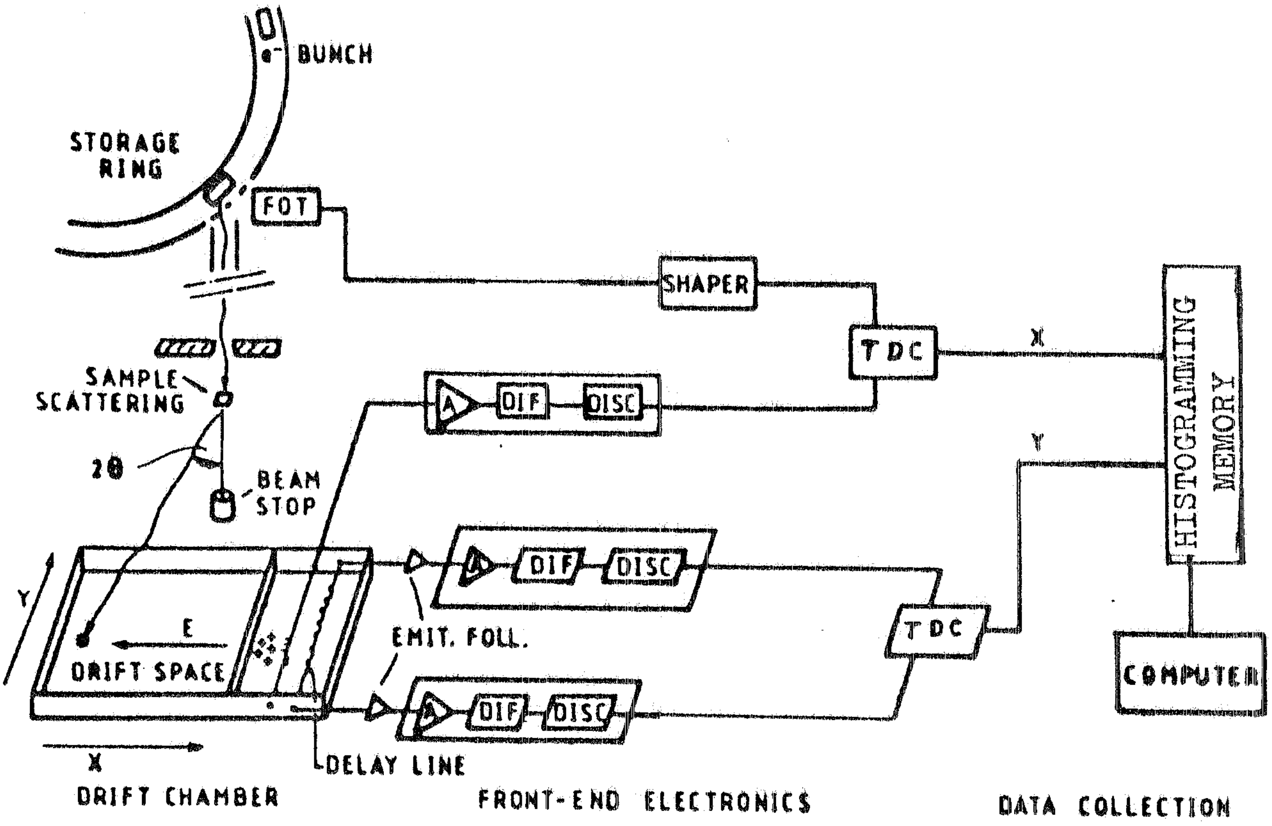

Fig. 3: Two-dimensional drift chamber detector and data acquisition system.

The electrons that are produced by absorption of the incident photon, by the gas mixture at a time t_0 in the position P(x,y), travel toward the anode, under the influence of a constant electric field E with a constant velocity v. The signal is received at the anode at the time t_1, when the avalanche of secondary electrons is produced. The coordinate x of P(x,y) is given by $x = v(t_1 - t_0)$. In general with a conventional X-ray source we cannot measure the time t_0 because the X-ray photon is totally absorbed by the gas in the chamber. But when the source is a pulsed synchrotron radiation beam, we can detect a signal at time t_0 from radiation flashes emitted by the same source(4).

This light flashes are actually emitted with the same time-structure as electron-bunches circulating in the storage ring. Obviously, the position of the photons originating from a given bunch on the detector area can be correctly determined by using the reference time t_0 associated with the same bunch.

The maximum drift time $(t_1 - t_0)$ along the whole length of the chamber must be less than the bunch periodicity. The time length of the bunch instead gives us the accuracy of the measurement of t_0. The time structure of European Machine in the single mode operation will have a bunch length of 4x41 psec with a periodicity of 2.6 μsec. With a drift velocity of 50 μm/nsec in detector, using Argon mixture at pressure of 1 bar, the maximum length permitted is 130 mm. The accuracy, which we can have in the localization of the position of the converted photon in the gas, is determined by range of photoelectron in the gas, by the variance of thermal space diffusion of electron swarm and by length of the bunch.

For X- ray at $\lambda = 1.54$ Å the maximum range of photoelectron is 180 μm in Argon mixture, the variance of thermal diffusion of electrons is 22 μm for 10 mm of the drift length and 87 μm for 130 mm of detecting area(5). Indetermination due to length of the bunch is 8 μm. From this data we obtain an estimation of spatial resolution of 200 μm, comparable to that obtainable at Frascati of 170 μm, and an angular resolution of 10^{-4} rad (20 seconds of arc) at 2,000 mm from the sample.

The total angle covered by detector should be instead of 6.5×10^{-2} rad (3.72°).

In the case of ESRF Machine it is possible to operate with three bunches of electrons circulating in the storage ring. In such a case the periodicity of the bunch is 886 nsec so the area of drift chamber has to be reduced by a total window at 43 mm in the drift direction. This area corresponds a total angle of detecting of 2.5×10^{-2} rad (1.33°) when it is 2,000 mm from the sample. This condition of operating increases the count rate up to 1 MHz (also in two-dimensional detection mode).

The positional coordinate y of the incident photon is obtained by the time difference measured on a continuous delay-line of the signal induced from the anode. The delay-line is placed closely behind the anode. The precision of measured coordinate y is essentially determined by the range of the avalanche discharge producing the signal of the anode wire and by electric characteristics of the delay-line. Actually with a continuous delay-line, operating at Frascati, a resolution better than 200 µm has been obtained using the Argon mixture. The characteristic of this delay-line is delay of 11.5 nsec/mm, good because it allows the use of cheap electronics.

The time drift signals and the delay line signals can be read out by two fast encoding time interval metres (TDC) and sent to a computer by a suitable histogramming memory to obtain a three-dimensional imaging of diffraction patterns. This diagram represents the spot position in the xy plain and the recording counts along the z-axis(6).

With a pixel size of 4×10^{-2} mm^2 a detector of area 130x130 mm^2 will have 422,500 pixels, while one of area 43x43 mm^2 should have 55,200 pixels.

Because the efficiency of the detector depends on the gas mixture used, the Argon mixture in a detector with 10 mm of gap has an efficiency of 17% at $\lambda = 1.54$ Å.

Using Xe mixture at 1 bar of pressure the efficiency is 84%, but in this case the drift velocity should reduce to 25 µm/nsec.. The useful area detector is reduced to 65x65 mm^2 with storage ring operating in the single bunch mode and to 21.5x21.5 mm^2 in the three-bunch mode. The spatial resolution should be better than 70 μm in both coordinates. Because the pixel size is 3.8x10^{-3} mm^2, the detector should have about 1,100,000 pixels or 120,000 pixels respectively. In such a case it is good to reduce the maximum distance sample-detector to 1,000 mm or less to have comparable figures on angular detection with detector using Argon mixture.

The Fig. 4 reports significant figures of the proposed detector using Argon mixture to other devices normally used.

In table I we summarize the characteristics of SAXS camera.

TABLE I - Parameters of the SAXS Camera in its longer distance configuration.

Cross section of pin-hole collimated primary beam	0.2	mm^2
Primary beam angular divergence (2α)	3.7	sec. of arc
Maximum angle covered by the beam-stopper	105	sec. of arc
Angle covered by residual parassitic scattering (2θ)	124	sec. of arc
Minimum scattering vector h at λ = 1.54 Å	2.5x10^{-3}	Å^{-1}
Maximum diameter of scattering unit d	2500	Å
Power incident on specimen at λ = 1.54 Å, with $\Delta\lambda/\lambda = 10^{-4}$	10^{13}	phot/sec.
Quantum efficiency of detector operating in Argon flow at λ = 1.54 Å	17%	
Detector size: in single bunch operation	130x130	mm^2
in three bunch operation	43x43	mm^2
Total angle subtended by the detector	3.72°	
Angular resolution in the plane of the detector:		
x-direction	20	sec. of arc
y-direction	20	sec. of arc
Pixel size	4x10^{-2}	mm^2
Number of detector pixels: 1. case	422,500	
2. case	55,200	
Maximum count rate per pixel	10^{-4}	cps
Maximum counting capability of detector	10^{-6}	cps

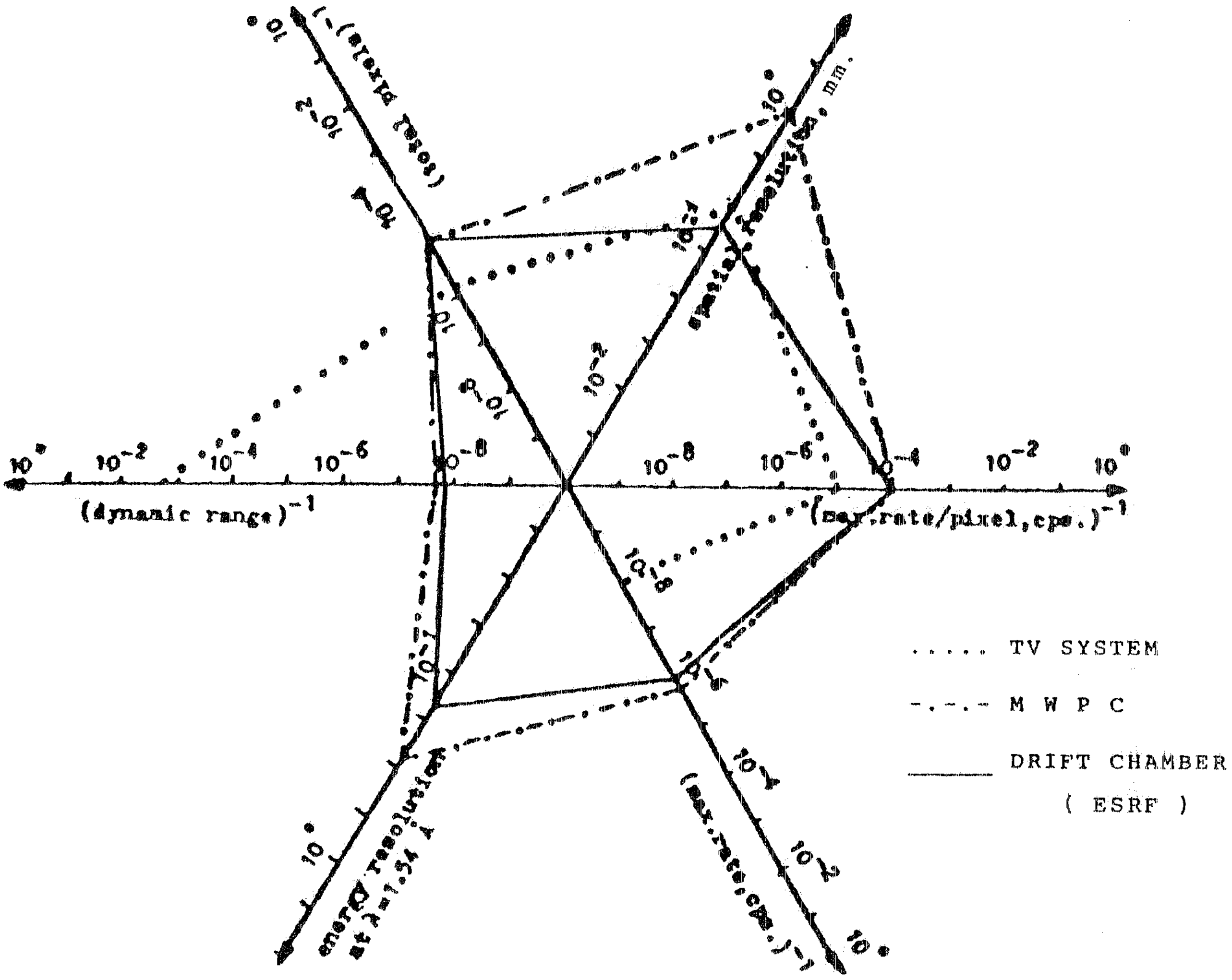

FIG. 4: Diagram of the significant figures of the proposed detector using Argon mixture and operating with three-bunch periodicity comparable to the usually devices.

CONCLUSION

The use of the scattering camera with the drift chamber described above will allow high precision measurements of anisotropic ultra-small angle scatterig from small samples.

The detector is flexible because it can use other gas mixture and detecting area can be varied. For good performances a relative short geometry of apparatus is required.

The apparatus will be built and partially tested at Frascati Synchrotron Radiation Facility by our group and by groups of the University of Bristol (U.K.) and the University of Bologna (I), in conjunction with technicians and Synchrotron Radiation experts working under ESRF contract.

Services and workshops of INFN-LNF will be used. The time realization of the apparatus will be estimated as soon as the number of collaborators is fixed.

The total cost is valuable in about 210 kECU, without monochromator and computer, as specified in the following Table:

TABLE: COSTING

- Collimated system	15 kECU
- Sample-holder chamber	20
- Optical table	12
- Stepping-motors	31
- Detector	15
- Apparatus for Ar and Xe gas mixture	62
- Electronic of detecting system	55
TOTAL	210 kECU

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