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Referat (sammandrag)

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PERFORMANCE OF AN ON-DEMAND BEAM EXCITATION SYSTEM FOR PIXE ANALYSIS

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A system for electrostatic on-demand beam excitation in PIXE analysis has been built and carefully tested. Significantly increased count rate capability without any need for corrections for electronic dead time and with less target deterioration makes the system superior to a traditional system with electronic pile-up rejection. By adding a fast external electronic pulse pile-up rejection system, a pile-up interv l of about 250 ns is obtained. The detailed behaviour of the particle beam during deflection and experience from using the system in routine analysis are reported.

1. INTRODUCTION

Particle Induced X-ray Emission, PIXE, was introduced in 1970[1]. The method is today used in many laboratories and has been applied to a variety of analytical problems[2].

For PIXE-analysis, electrostatic accelerators or cyclotrons are normally used to produce charged particles with energy in the interval from 1 to 5 MeV/u. When these particles impinge on a target they create inner shell vacancies in the target atoms. When such a vacancy is refilled the excess energy may be emitted as a characteristic X-ray. The X-rays are normally detected by an energy dispersive Si(Li)-detector with high energy resolution.

Normal beam intensities in PIXE analysis are in the interval from 1 to 200 nA. To reduce the analysing-time, high beam intensity is normally used. The electronics used for detector pulse processing will, however, limit the number of pulses processed per unit time. Too high count rate can cause severe distortion of the pulse-height spectrum. Peak centroid shifts and peak broadening will occur in the spectrum as will pulse pile-up effects. Evaluation of X-ray spectra is normally made by computer codes which are sensitive to spectrum distortions. In addition, the particle beam intensity is restricted by target deterioration, due to dissipation of energy as the charged particles slow down in the matrix. Some samples, e.g. biological tissues, are particularly sensitive to high beam intensities.

2. PILE-UP REDUCTION IN ENERGY DISPERSIVE X-RAY ANALYSIS

2.1. X-ray pulse processing

After detection of an X-ray in the Si(Li) crystal, a pulse proportional to the X-ray energy is delivered from the preamplifier to the main amplifier. A conventional X-ray main amplifier uses pulse shaping time constants of between 2 and 10 µs, giving pulse processing times in the interval of 20 to 100 µs. The probability for pulse pile-up effects due to the overlapping of X-ray pulses is proportional to the pulse processing time. In a fictitious X-ray energy spectrum with one single X-ray line, pulse pile-up will appear as a continuum starting just above the full energy peak and ending in a peak at double the energy of the X-ray peak (compare fig.3a). The continuum is due to pulse overlapping when two pulses arrive with a relatively large time difference but still close enough to be added partly in the amplifier. Such a continuum acts as an increased background for other possible peaks in the energy interval in question, thus significantly increasing the lower limit of detection. The discrete pile-up peaks, emanating from pulses very close in time, will interfere with X-ray peaks in their immediate vicinities. Pulse pile-up can be suppressed by the introduction of an electronic pile-up rejector[3].

X-ray main amplifiers are more or less sensitive to high count rates which can cause X-ray peak broadening and peak centroid shifts due to incomplete base line restoration. The X-ray spectrum will be distorted by such effects as well as by the pulse pile-up effects. Even very slight changes in the spectrum may introduce gross errors in the quantitative evaluation. The latter is normally made by automatic or semi-automatic computer codes which fit the X-ray spectra. Since computer codes are normally quite sensitive to changes in energy resolution and peak centroid position values, such spectral distortion causes the failure of automatic spectrum evaluation procedures and, since no PIXE laboratory running on a routine basis can avoid the use of computer evaluation of X-ray spectra, there is an obvious need for suppressing them.

2.3. Electronic pile-up rejector

The electronic pulse pile-up rejector (PPR) includes a fast amplifier parallel to the slow, high-resolution spectroscopy amplifier. The PPR senses if two consecutive events from the preamplifier overlap in time, normally with a pulse pair resolution of 0.5 to 1 µs. If two events overlap, both are rejected by inhibiting the main amplifier output, thus almost completely eliminating the pile-up continuum. To enable a quantitative analysis, the electronic deadtime thus introduced has to be corrected for.

The discriminator of the PPR is set at a threshold level, small enough to include the pile-up emanating from X-rays of the lowest energy of interest. Noise, however, should not trigger the PPR since this would introduce unnecessary electronic dead-time.

The time interval in which pulse pile-up due to the finite time resolution of the electronics cannot be detected is called the pile-up interval τ. Any two pulses which arrive within this interval are not rejected by the PPR and occur as one peak at an energy approximately equal to sum of the two X-ray energies referring to the two pulses. Knowing the number of pile-up events per unit time, τ can be derived from Poisson statistics [3] giving the expression :
$N_{ij}=(\tau_{ij}+\tau_{ji})\cdot N_i \cdot N_j$, $i \neq j$ (A), where N_i is the number of events with energy E_i per unit live time, N_{ij} the number of pile-up events emanating from the combination of E_i- and E_j- events per unit live time and τ_{ij} the pile-up interval between pulses from X-rays of energy E_i and E_j. For double energy peaks ($E_{ii}= E_i + E_i$) this expression becomes:
$N_{ii} = \tau_{ii} \cdot N^2$ (B). The formulae show that the number of pile-up events will increase as the square of the count rate. For very high count rates there will also be a significant probability for triple pile-up (compare fig.3b).

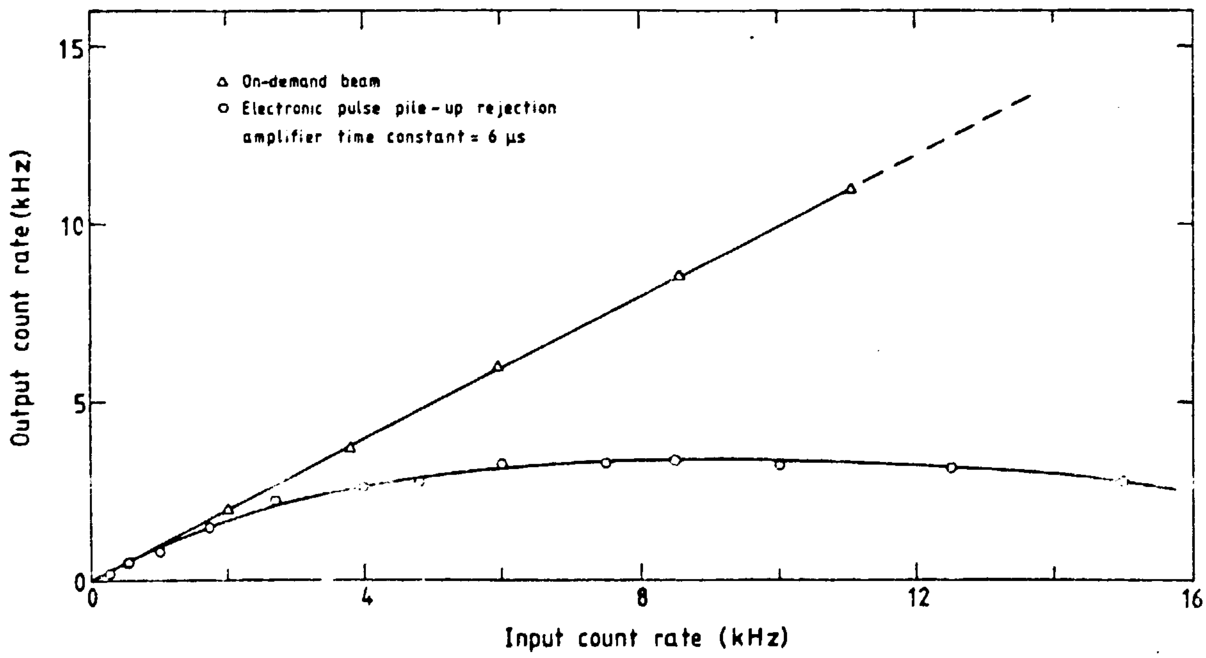

Fig 1. Effective count rate (counts per unit real time) in the X-ray spectrum versus the input count rate for on-demand beam irradiation (Δ) and electronic pile-up rejection (o) respectively. The results were obtained during irradiation of a thick copper plate.

With an electronic PPR, an increased input count rate does not always imply an increased output rate. Instead, a maximum output count rate, determined by the pulse shaping time constant, will be reached for a certain input count rate (fig.1). If the count rate is high enough, the deadtime will eventually reach 100% and no output will occur from the amplifier. The system is paralyzed.

2.3. Pile-up reduction by on-demand beam excitation

A different technique of pulse pile-up reduction makes use of an on-demand excitation system[4,5]. When an event is detected at the output of the preamplifier, the excitation source is shut off. The source is then kept off during the pulse processing time. When the amplifier is ready to accept and to process another pulse the excitation source is turned on again. In this way a non-paralyzable system is obtained.

In particle induced X-ray analysis a suitable way to shut off the excitation source is by the insertion of two parallel electrostatic deflection plates in the beam transport tube[5], between which the charged particles pass on their way towards the sample. When an X-ray event has been detected, a transversal electrical field is applied between the plates forcing the charged particles to deflect and thus removing the particle beam from the sample. The electric field is switched off once the event has been processed in the amplifier.

If a system for fast leading edge discrimination is used to detect the X-ray signal from the preamplifier the beam may be turned off within a very short time. The pulse pile-up interval is determined by the sum of the times for the leading edge discriminator to detect the pulse, for the signal to reach the beam deflection circuit, to deflect the beam and the transit time for the last undeflected particles to move from the deflection plates to the target.

3. ON-DEMAND BEAM SYSTEM

An on-demand beam excitation system has been implemented in the beam line of the PIXE-facility at the Lund Institute of Technology[6]. The electronics of the system is similar to the one presented in ref.[5] which is referred to for details of electronic design. However, the discharge time of the deflection plates is shorter in the system discussed here.

3.1. Technical description

A pair of deflection plates (0.46 m x 0.04 m) made of aluminium are mounted in parallel with a spacing of 8 mm, in one of our standard beam transport tubes (diam.:0.06 m). The distance between the centre of the beam deflection system and the target is 1.0 m. A beam collimator (diam.:3 mm) is placed 0.6 m from the target. When deflected, the beam hits the collimator and no particles reach the sample.

The deflection is controlled by the X-ray signals from the preamplifier (see fig.2). This is a pulsed optical feedback preamplifier (Kevex 2003) producing step signals proportional to the X-ray energies detected, thus forming an increasing ramp voltage signal. When the DC-level reaches +5V the preamplifier is reset by an optical signal from a light emitting diode. An emitter follower stage, necessary to match impedances, differentiates the preamplifier signal, which is then amplified by a fast timing filter amplifier (ORTEC 474) and fed into a single channel analyzer (ORTEC 406A), the output signal of which (LLD output, +5V, 500 ns wide) triggers the control electronics (TU) of the deflection system. The control electronics provides a 95 V positive going pulse to open a power tube (PL 519) for the grounding of one of the deflector plates. These plates are normally both maintained at a voltage of 1-2 kV. The 10% to 90% rise time of the electric field between the plates from zero to 1200 V/cm is 35 ns.

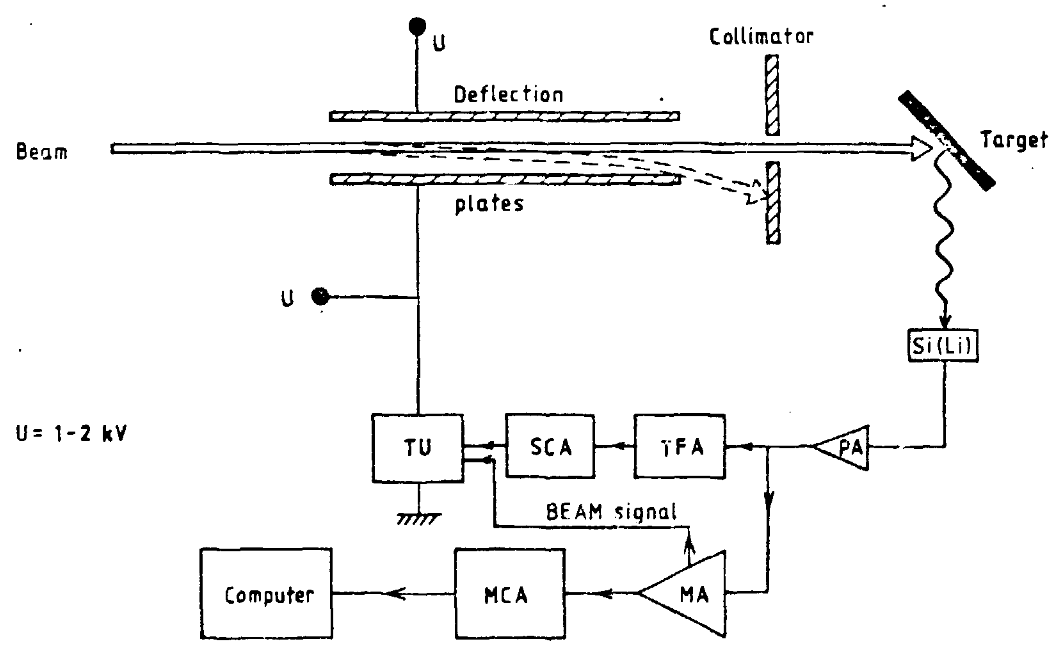

Fig 2. Block diagram of the electronics used for on-demand beam pulsing. PA = preamplifier, MA = main amplifier, MCA = multi-channel analyser, TFA = timing filter amplifier, SCA = single channel analyser, TU = trigger unit and BEAM signal = logical output from main amplifier during the processing of a pulse.

The deflection duration can be set on the control electronics or be controlled by the BEAM-signal from the amplifier. This signal shows that the amplifier is busy processing an event and the deflection is maintained also during the long busy intervals (0.5–2 μs) following a pulsed optical reset in the preamplifier.

3.2. Pile-up interval

During the detection of an X-ray event, the charge collection time in the Si(Li)-detector is typically around 50 ns[3]. The measured delay of the signal during its transmission from the preamplifier to the power tube is about 300 ns. For the projectiles normally used in this laboratory (protons, 2.55 MeV), the transit time from the plates to the target is about 40 ns. When bombarding a thick homogeneous Cu-plate with different count rates (1500–5000 cps) the double energy pile-up peak for the K_α-line has been measured and from formula (B) has been calculated to be 400±20 ns (± one standard error of the mean). This is in fair agreement with the measured time delays. In fig 3 are shown the spectra from bombarding this target with a) no PPR, b) with PPR, c) with on-demand beam irradiation and d) with simultaneous use of PPR and on-demand beam irradiation respectively (the total effective count rate in each spectrum is about 4500 cps).

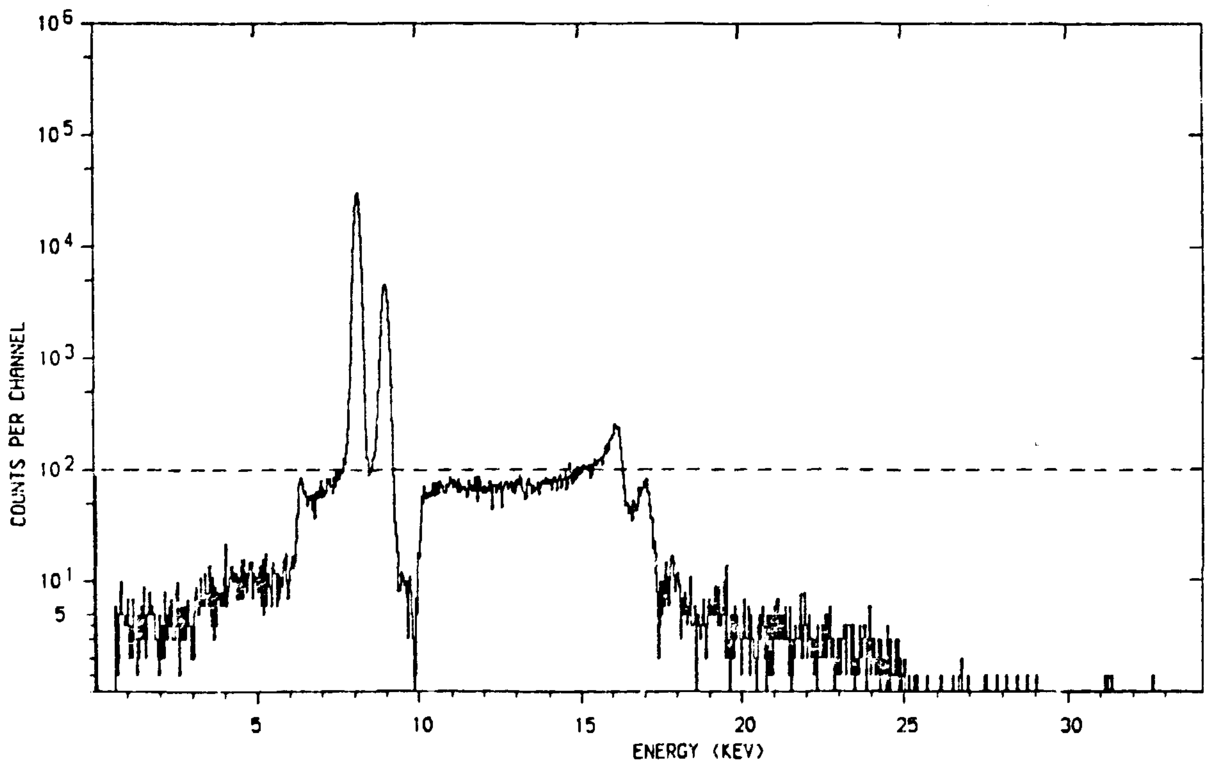

Fig 3. The spectra obtained during equally long bombardments of a thick copper plate using a 340 μm Mylar X-ray absorber and a amplifier time constant of 4 μs. The effective total count rates are in all cases about 4500 cps (real time). The broken lines shown at 100 counts per channel are given to facilitate comparison between spectra. The spectra refer to the following conditions: a) no pile-up rejection

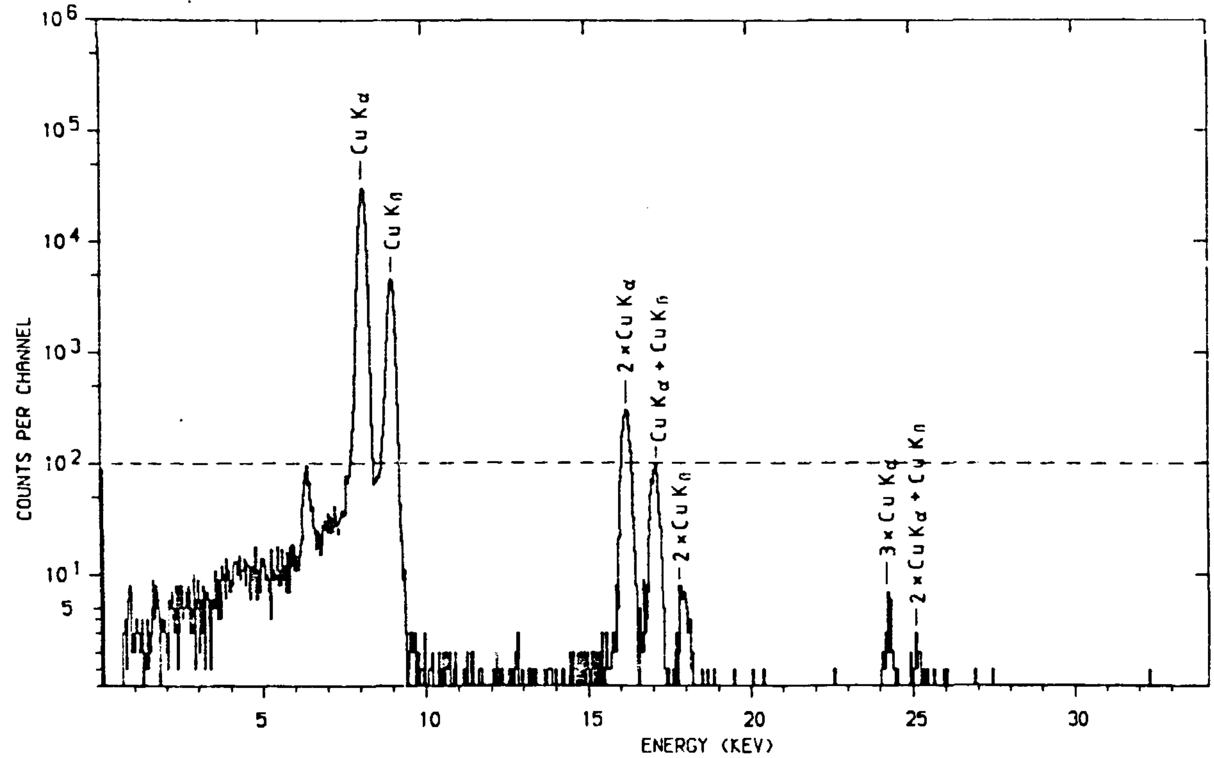

Fig 3 b) electronic pulse pile-up rejection

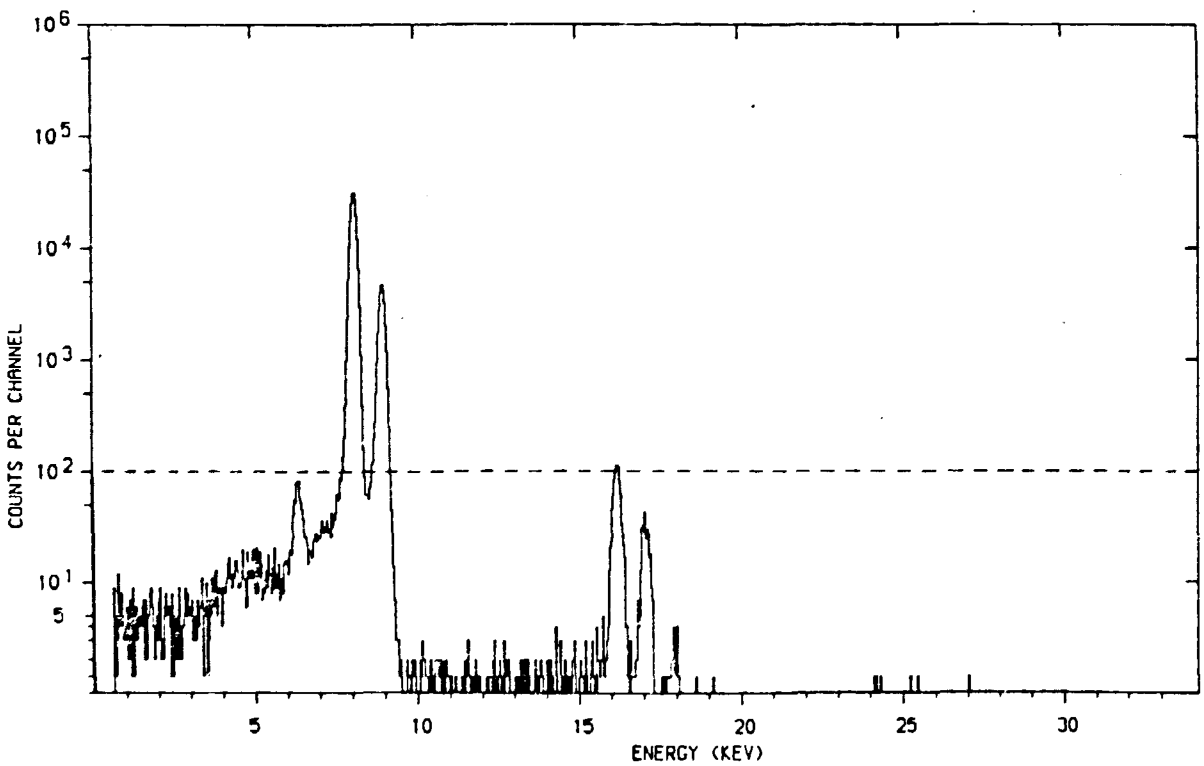

Fig 3 c) on-demand beam irradiation

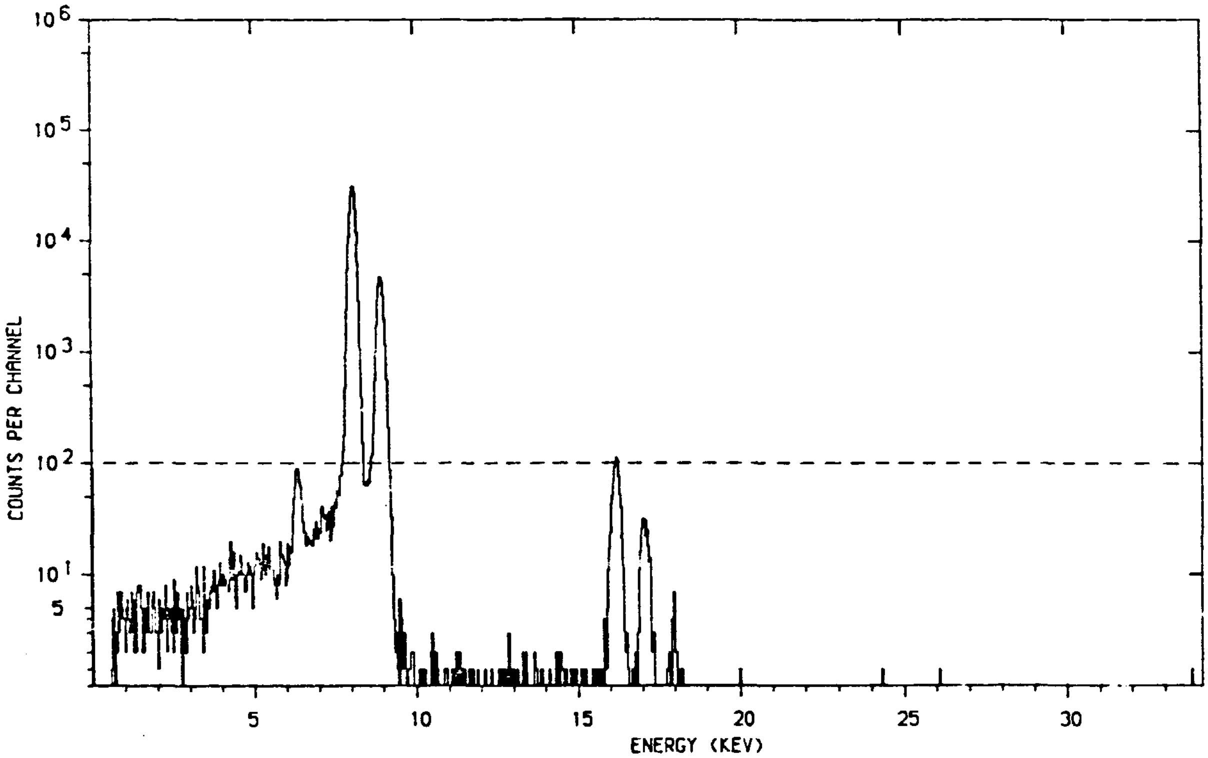

Fig 3 d) the simultaneous use of on-demand beam irradiation and electronic pile-up rejection

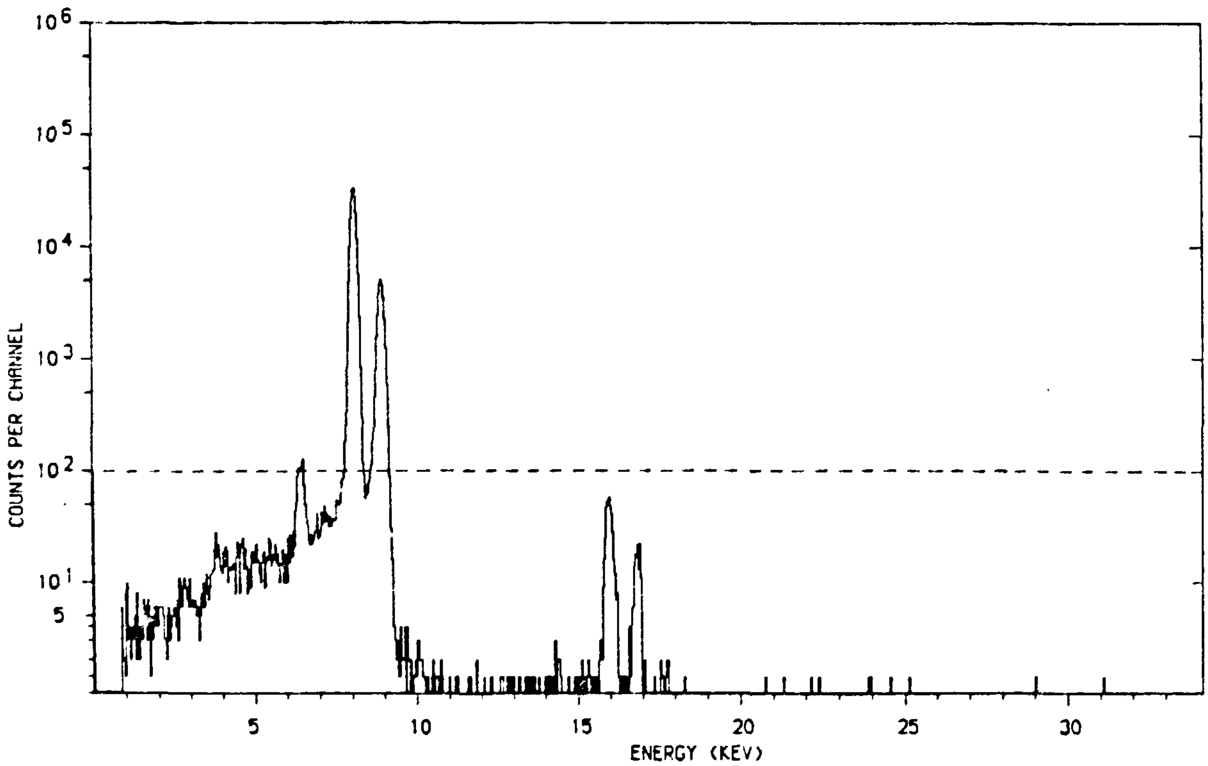

Fig 3 e) similar to d) with addition of a fast external PPR.

In fig. 3 b), a drastic suppression of the continuous pulse pile-up can be observed with an electronic PPR as compared to fig. 3 a). In c) the use of on-demand irradiation is superior to PPR in reducing double pile-up peaks and in that no triple pile-up peaks are observed. The simultaneous use of on-demand irradiation and PPR (fig. 3 d) gives a slightly lower pile-up continuum than in c). If no PPR is used when the beam is deflected during the processing of an X-ray event and hence no electronic inspection is performed, another event, e.g. caused by particles being scattered out of the deflected beam into the normal beam path and thus reaching the sample, can be registered during the off-beam period thus interfering with the event being processed and causing pile-up in the continuum region. In the analytical set-up reported, the commercially available PPR in the X-ray main amplifier is used together with on-demand beam irradiation.

It is possible to shorten the pile-up interval in on-demand beam irradiation if commercial modules with smaller signal delays are used for leading edge discrimination and by reducing the signal delay in the control electronics (TU in fig. 2). We estimate that in this way it would be possible with this on-demand beam system to reach a pile-up interval of about 200 ns.

For protons with energies in the interval of 1 to 4 MeV and for alpha particles from 3 to 9 MeV, the variation in the pile-up interval will be less than 10%, which is insignificant for practical analytical purposes.

In commercial PPR:s the manufacturers, due to the need to compromise between detecting pulse pile-up from low energy X-rays and suppression of electronic noise[7], limit the pulse pair resolution to about 500 to 1000 ns. It is, however, possible to obtain a pulse pair resolution of less than 300 ns by using commercial NIM modules.

In fig 4 is shown a block diagram of the electronics which have been used for external pulse pile-up rejection. A time-to-pulse-height-converter (TPHC, Ortec 467) has been used to measure the time intervals between successive X-ray events and if two events occur within a preset time interval (TPHC time interval), a pulse will be produced and the TPHC is then ready for a new measurement. The negative signals delivered from the fast discriminator (FD) upon the detection of an X-ray event are used to stop and with a delay (10 ns) to start the TPHC. A +8V standard pulse (SCA in fig 4) with a width of 10 μs is produced as soon as a time interval has been measured and, after a suitable delay, it may be used to

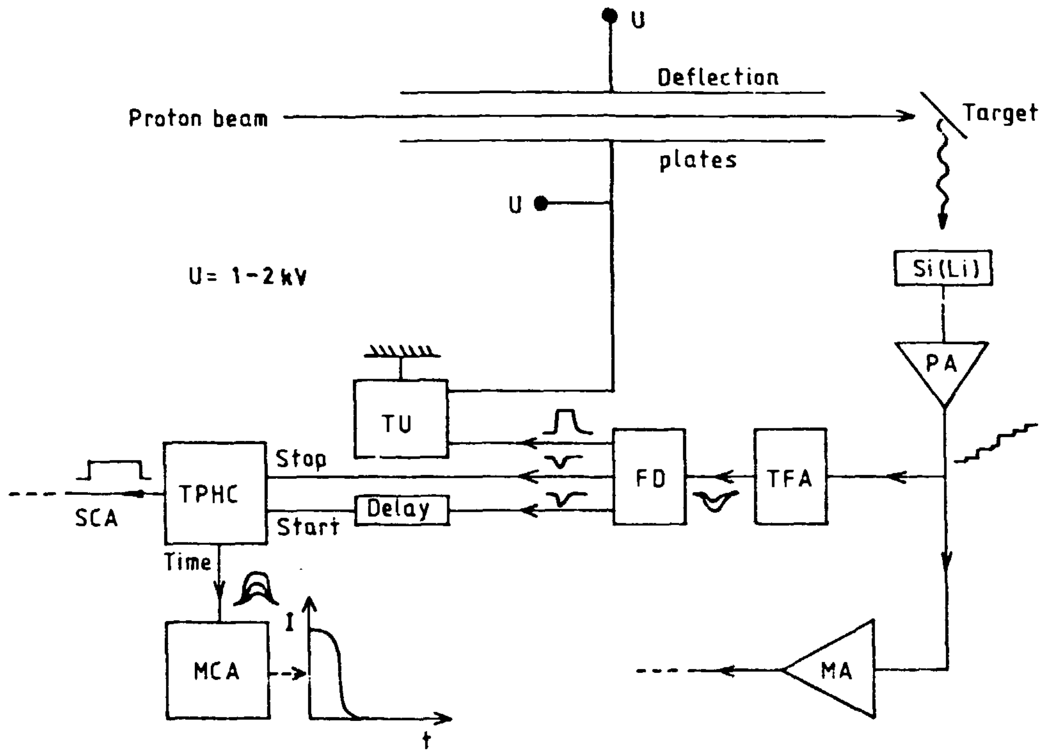

Fig 4. Block diagram of the electronics used for external pile-up rejection and when studying the detailed behaviour of the proton beam. A target is irradiated and the preamplifier signals enter a timing filter amplifier (TFA) followed by a fast discriminator (FD). The latter triggers the trigger unit (TU) and supplies a time-to-pulse-height-converter (TPHC) with both start (with a suitable delay) and stop signals.

inhibit the input of the ADC thereby rejecting the X-ray events which would otherwise be found as pile-up in the spectrum. If a TPHC interval of 2 μs is chosen ideally all pulses less than 2 μs from one another will be rejected. In this way a pile-up interval of about 250 ns is obtained. The ultimate limit of such an electronic rejection system, able to distinguish between two adjacent pulses, will be set by the charge collection time in the Si(Li) crystal (see sect. 3.2) and the pulse pair resolution of the electronics. The rise time of the X-ray pulses from the preamplifier has been measured to be about 100 ns. Used together with an on-demand beam system it is then possible to achieve a reduction of the intensity of the double energy peaks as compared to pure on-demand beam irradiation without introducing any significant dead-time ($< 1\%$). In fig 3e is shown a spectrum similar to 3d but with the addition of the electronic pulse pile-up rejection system described.

To account for the remaining pile-up peaks, which will still be significant at high count rates, the spectrum evaluation computer code in use has been modified to facilitate accurate corrections for pile-up peaks .

3.3. Routine applications

The on-demand beam system described is in routine used and works very well requiring only minor maintenance. The practical count rate is limited to about 10 000 cps (with an amplifier time constant of 6 µs), primarily by the limited power capacity of the deflection tube. This is a common electron beam deflection tube used in many commercial colour television sets and thus readily available at a low cost. It could, however, be replaced by other types of tubes capable of withstanding much higher power dissipations, e.g. high power transmitting tubes.

3.4. Performance of the on-demand beam system

Extensive work has been carried out to test the behaviour of the deflection system in detail and to check whether it is reliable in conjunction with quantitative PIXE-analysis. The tests normally described in the literature[5,9] , imply the comparison of X-ray spectra with electronic pile-up rejection with those obtained with an on-demand beam system (fig. 3). It is, however, also important to study the detailed behaviour of the particle beam during deflection and return.

3.4.1. Deflection and return of the beam

When an X-ray event is detected, the signal is sensed by the on-demand electronics. A pulse then opens the power tube to ground one of the deflection plates and thus deflect the beam. The removal of the beam from the target should occur as rapidly as possible. Similarily to many other laboratories, in the Lund PIXE arrangement we use a diffuser foil (1 mg/cm^2 gold foil) to obtain a homogeneous beam at the target[6]. The foil scatters the particles in the beam away from their initial directions. When deflection takes place, the beam is moving transversally and the particles will eventually hit the collimator situated just in front of the gold foil. Due to the scattering in the collimator edges and the foil, some particles may hit the sample and produce X-rays during and after the beam removal. If these X-rays reach the detector, they cause pulse pile-up in the electronics.

To study the detailed performance of the system the TPHC (see fig. 4) has been used to measure the time interval between two successive X-ray events when the beam deflection is active. In addition to the +8V pulse (see sect. 3.2) an analogue signal with an amplitude proportional to the measured time interval is produced subsequent to each time measurement. The TPHC is started by an X-ray signal after a suitable time delay and stopped by the successive X-ray signal. By selecting the delay of the start signal properly (about 250 ns), it is possible to start the time measurement just before the deflection of the beam starts. As the deflection proceeds, the probability for the occurrence of a new event will decrease. Hence, with repeated beam deflections, the frequency of stop signals from a succesive event is expected to decrease with the time after beam removal. This can be demonstrated by storing the measured time intervals as a time spectrum in a multichannel analyser.

Figure 5 shows a time spectrum obtained during a 10 min irradiation of a Cu-target with a deflection frequency of about 5 kHz. The TPHC time interval is set to 0.4 μs in the left-hand part of fig.5 and to 80 μs in the right-hand. The diagram shows the time dependence of the count-rate when the deflection takes place. The 90% to 10% fall time is about 25 ns. The right-hand part of the figure shows the behaviour of the beam when the deflection duration is set on the control electronics to 65 μs. The beam returns to the target within 64 μs and the beam intensity then recovers with a 10% to 90 % rise time of 2.1 μs. The longer time for beam return is due to the longer time required to return the grounded plate to its initial potential than to perform a sudden grounding.

3.4.2. Beam charge integration

Since the time required to remove the beam from the target is very short compared to the pulse processing time ($< 1\%$), no dead-time corrections have to be made. However, the deflection system (power tube, plates, etc) will act as a strong source of electromagnetic radiation in the radiofrequency band. The current measurement system, including irradiation chamber, coaxial cables and Faraday cup, forms an antenna, picking up the radiation. The currents which may be induced (normally below a few nA) can cause significant inaccuracies when low beam currents ($I < 10$nA) are used. The rather large irradiation chamber used in this particular experimental arrangement may be part of the explanation of this phenomenon. Careful shielding and grounding eliminate or suppress these currents but the attention of presumptive users of such systems should be drawn to the

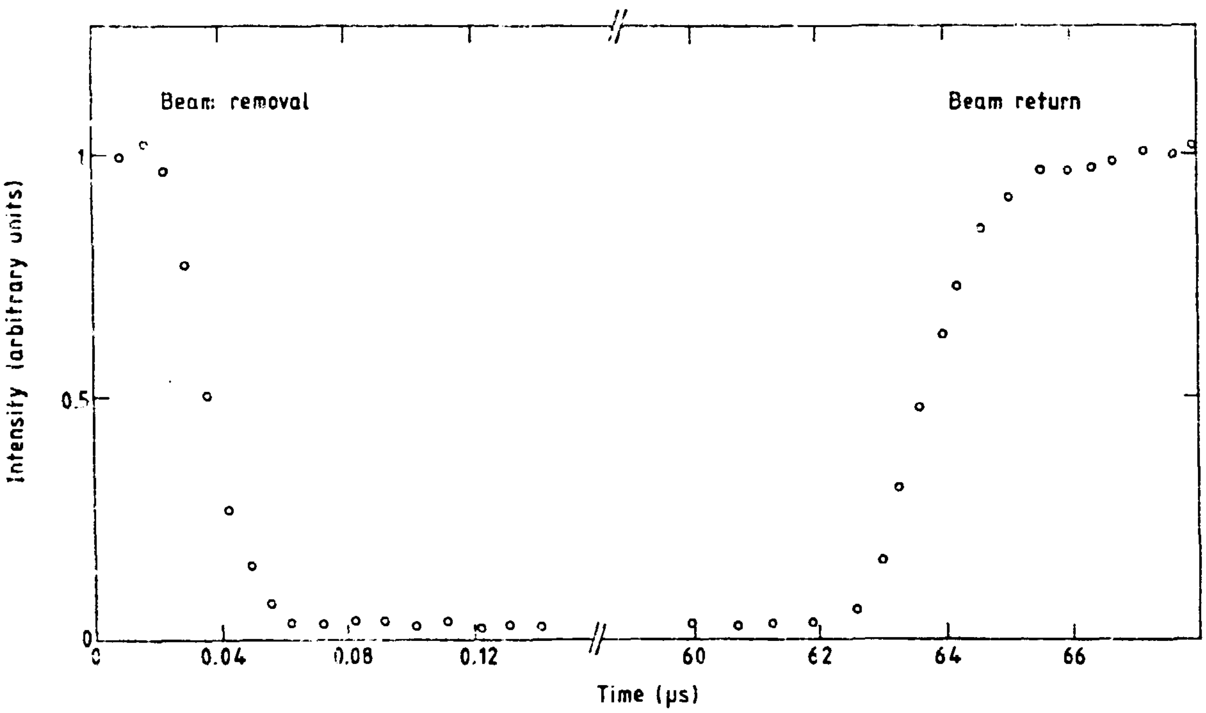

Fig 5. Time spectra from a TPHC with TPHC time interval of 400 ns (left) and of 80 μs (right) obtained during the irradiation of a thick copper plate with a deflection frequency (cps) of about 5 kHz.

problem, in order to avoid analytical inaccuracies when low beam currents are used.

The use of a collimator to stop the deflected beam requires careful alignment of the beam, i.e. the beam must pass straight through the collimator when no deflection takes place. If the main part of the beam hits one side of the collimator but part of the beam is still let through the central hole, it will hit the sample and produce X-rays thus triggering the beam deflection. During the pulse processing, when no particles are expected to reach the sample, part of the beam is steered into the collimator hole, thus giving a residual beam intensity at the sample and causing pulse pile-up and erroneous charge integration. This situation may be detected by repeated analyses of a well-known reference standard sample[10] and can be avoided by careful initial alignment of the particle beam through the collimator.

4 SUMMARY

Compared to the electronic PPR, an on-demand pile-up reduction system has several advantages. Since the excitation source is shut off almost immediately after an event is detected, there is no need for any dead-time corrections in quantitative analysis and the system cannot be paralyzed. Compared to a conventional PPR, the pile-up peaks in the X-ray spectrum will be significantly reduced for the same effective count rate (cps, real time) as can be seen from fig.3.

Furthermore, since the output rate of an on-demand system will be equal to the input rate (see fig.1), fewer count rate problems will occur. High output count rates can be achieved without the occurrence of any significant peak centroid shifts or peak broadening since the amplifier will be able to perform adequate base-line restoration for each individual event.

Target deterioration due to heating is dependent on beam intensity and deterioration due to radiation damage proportional to the total integrated beam charge (accumulated radiation dose). Being without any dead-time losses, the on-demand pulsed beam excitation system enables the use of lower currents and less charge for the same amount of information. For samples which are sensitive to radiation damage or heating, e.g. biological samples or samples containing volatile compounds, the use of on-demand beam excitation shall prove especially advantageous.

A detailed study of the deflection process shows that the beam intensity falls off very rapidly as soon as the control signal reaches the electronic tube which grounds the one plate and also recovers acceptably quickly when the high voltage is reapplied to the plate. The total delay of signals from detection of X-ray events in the detector crystal to turn-on of the triggering unit, which grounds the plate is about 300 ns in our experimental arrangement but can be further improved by selecting electronics with shorter pulse delays.

A fast external pulse pile-up rejector was designed using standard NIM modules and, with the combined use of the on-demand beam excitation and the external PPR a pile-up interval of about 250 ns has been obtained.

It is our experience that minor problems may arise in the use of on-demand beam electronics since extra currents can be induced in the charge measurement system (especially for large volume chambers). These problems can, however, be overcome by careful shielding and grounding. To avoid increased pile-up and inaccuracies in quantitative determinations, careful alignment of the particle beam is recommended.

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