A NEW IXX SYSTEM AND ITS APPLICATIONS

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ABSTRACT

IXX (or PIXE－induced XRF) technique gains two main advantages over conventional PIXE method. First, it can be used to avoid or significantly reduce background and spectral interferences from major elements in the sample by proper selecting the primary target. Second, target damage is greatly reduced, so that it is more suitable for the analysis of heatsensitive and delicate specimens. A new IXX system with a very tight geometry is described. Some of its performances and preliminary applications are presented.

Key words: PIXE XRF IXX Matrix effect Target damage

I. INTRODUCTION

Particle induces X－ray emission (PIXE) as a useful analytical technique has been widely applied in many fields with its advantages of rapidity, sensitivity and multielemental analysis. However, PIXE has its limitations as well [1-2]. For example, the matrix effect, i.e. the spectral interferences from the major matrix elements of a sample hiders the good sensitivity for trace elements in heavy matrix specimens. And, the heating and radiation damage caused by particle bombardment limit accurate analysis of heat－sensitive and delicate specimens.

X－ray induced X－ray fluorescence technique can avoid such limitations[1]. Since Lin and co－workers did their work in 1978[3], IXX (or PIXE－induced XRF) technique has been gradually used to make up the drawbacks of PIXE based on a small accelerator[1-7]. Using this method, the best primary target can be chosen properly for each element analyzed to get the best detection sensitivity. So lighter constituents in heavy matrices can be analyzed effectively meanwhile the heating and radiation damage are minimized. Due to the secondary effect, the detection efficiency of IXX relative to incident protons is orders of magnitude less than that of PIXE. Therefore the beam current required in IXX must be at least $1-10\,\mu$A which can be provided from usual small accelerators. The X－ray fluence of the order of $10^{11}-10^{12}$ can be obtained and the minimum detection limit can reach 0.1－1 parts per million[1].

This paper reports some preliminary results of an IXX system based on a 4 MeV Van de Graaff accelerator. This system was transformed from an external PIXE system

and calibrated by using a set of spectroscopically pure thin foils. The trace elements in some medium and heavier matrix samples were analyzed more sensitively using this system compared with conventional PIXE. Moreover, it was employed in the analysis of paper-like samples.

II. EXPERIMENTAL

The experimental arrangement is shown in Fig.1. It is different from common design with a transmission geometry. The primary targets were pure metal foils sticked on

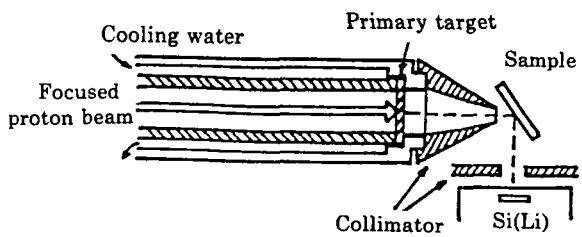

Fig.1 Schematic diagram of the apparatus arrangement of IXX system with a transimission geometry

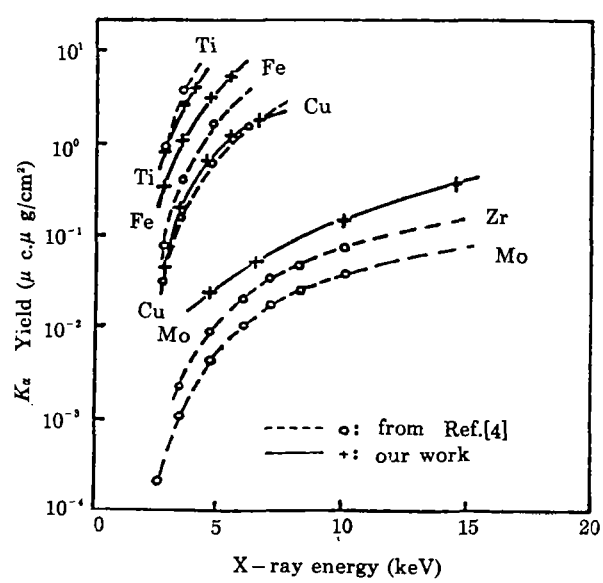

Fig.2 K yield calibration curves obtained using thin foil calibrators for primary targets: Ti, Fe, Cu and Mo, compared with the results in Ref.[4]

the exit window with strong vaccum glue instead of Kapton foil used in conventional external beam PIXE. A 2.3 MeV proton beam of 3 mm diameter from a 4 MeV Van de Graaff hits on the primary target. The primary X-rays generated by protons were collimated by a water-cooled collimator to a diameter of 4 mm and shielded

sufficiently. The primary target was isolated from the beam line and acted as a Faraday cup to monitor the beam current. The samples analyzed were placed at 45° to the direction of the incident X-rays. A Si(Li) detector was positioned at 45° to the sample. The counting rate of spectra was restricted to below 1 kHz to reduce the pulse pile-up.

In spite of the self-absorption effect, the X-ray yield of this system was no less than that of reflection geometry IXX system since very tight geometry and maximum solid angle of detection were adopted. The thickness of primary target of Mo, Cu, Fe and Ti were 35, 20,20, and 20 μ m, respectively. In order to stop the protons, a carbon layer of proper thickness was coated on the outer surface of the primary target. Calibration curves of this system were shown in Fig.2 and were compared with Ref. [4]. A set of Micromatter thin foils of known areal density ranging from 8-50 μ g/cm^2 was used in the calibration.

Ⅲ. APPLICATIONS

1. Determination of impurities in steel

The major component of steel is Fe which produces high intensity X-rays of 6.403 keV under energetic proton bombardment. The relatively high tail of Fe K_α peak hinders the measurement of Cr, V, Ti and etc, which determine the property of the steel. In addition, the high bremsstrahlung background for thick sample affects the sensitivity in the low energy region. Using a primary target of pure Fe, these elements can be excited sensitively by the primary X-rays. Meanwhile, Fe X-rays detected are only scattered X-rays from the primary target so that the interference to the other elements is greatly reduced.

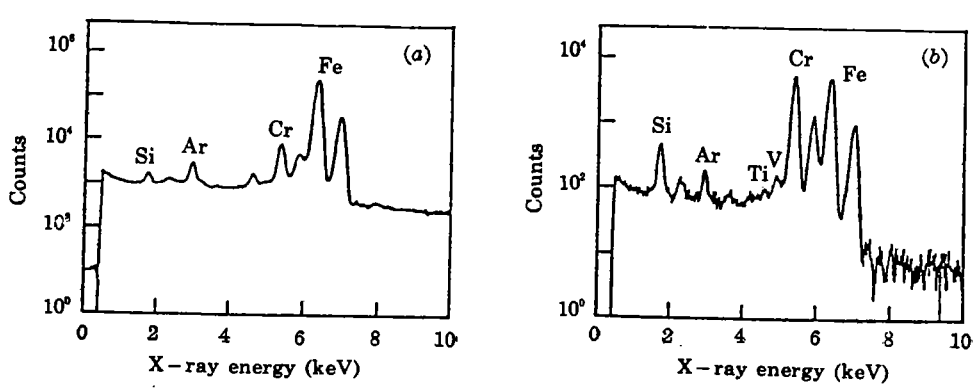

Fig.3 PIXE spectrum (a) and IXX spectrum (b) of a steel sample

(*a*) 2.3 MeV, 0.5 nA, 135° detection angle, 1200 s detection time

(*b*) primary target of Fe: 2.3 MeV, 1 μ A, 1200 s

A kind of steel sample was analyzed in our lab by PIXE and IXX respectively. The comparison between the spectra of two methods obtained is shown in Fig.3. The analyses of Cr, V, Ti, Ca and Si by IXX were obviously more sensitive than that by PIXE. The sensitivities for IXX were estimated at about 2 ppm for Cr, 5 ppm for V, 10ppm for Ti, 30ppm for Ca and 60 ppm for Si, while the proton beam current of 1 μ A was used.

2. Measurement of light calcium in Pb- Ca alloy

The poles of many storage batteries are made of Pb-Ca alloy. It is necessary to determine the concentration of light Ca element in alloy for examining its quality in the process of production. Fig.4 *(a)* gives a PIXE spectrum of a Pb-Ca alloy sample. It can be seen that high intensity X-rays of Pb (*M* serie) and bremsstrahlung background interfere the determination of calcium. The minimum detection limit of Ca for PIXE was above 100ppm. An IXX spectrum for the same sample is shown in Fig.4*(b)* using a primary target of Ti. Compared with PIXE spectrum, the sensitivity for Ca was much better. The MDL was estimated to be about 10 ppm while the proton beam current was 5 μ A for thick Pb-Ca target.

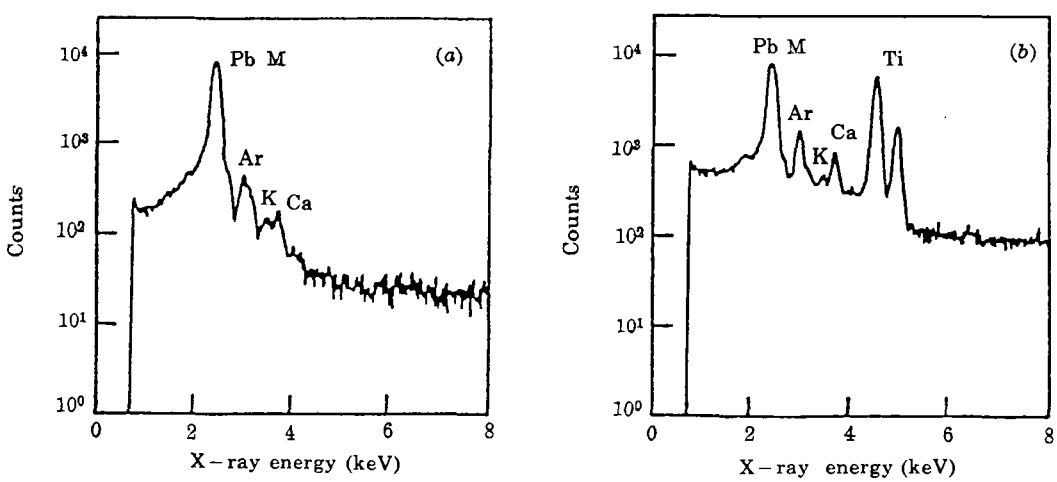

Fig 4 PIXE spectrum of Pb- Ca alloy sample(*a*) and IXX spectrum with a primary target of TI (*b*)

(*a*) 2.3 MeV, 0.5nA, 1200 s (*b*) 2.3 MeV, 5uA, 1200 s

3. Analysis of cobalt in excess nickel

In the PIXE analysis of light cobalt in excess nickel, the high intensity of Ni X-rays may interfere the measurement of Co. Fig.5*(a)* gives a PIXE spectrum obtained in the analysis of one liquid sample containing Ni and Co in ratios of up to 500: 1. As the *K* absorption edge of nickel is at 8.331 keV, the Cu K_α X-rays of 8.047 keV excite no nickel but cobalt atoms. Using a Cu primary target, an enhancement of

sensitivity by a factor 11 for Co was observed as shown in Fig.5 (*b*). In order to reduce the nickel X-rays induced by Cu K_β X-rays, an absorber foil of 30 μ m Ni was placed between the primary target and the sample. So that the interference from Ni to Co was almost completely excluded, as shown in Fig.5 (c). A sensitivity of 10ppm of Co in Ni was obtained.

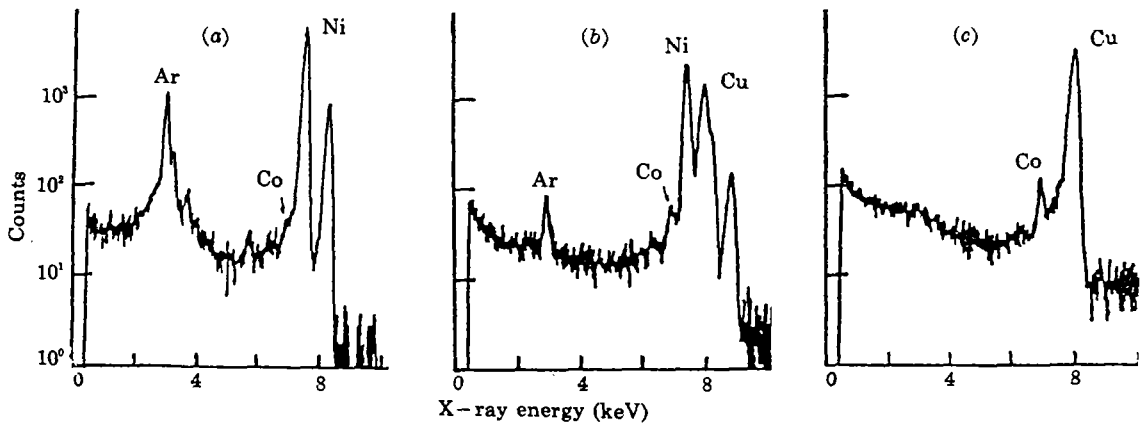

Fig.5 PIXE spectrum (*a*) and IXX spectrum (*b*,*c*) of a liquid sample containing escess Ni and Co

(*a*)2.3MeV, 0.2nA, 1200 s (*b*)with a primary target of Cu: 2.3 MeV, 0.5 μ A, 1200 s (*c*)with a 30 μ m Ni filter between primary target and sample

4. Nondestructive analysis of paper- like samples

Due to heating and radiation damage caused by proton bombardment, it is difficult to nondestructively analyze the heat-sensitive and delicate samples by PIXE, especially paper-like specimen. As shown in our previous work[8], both low beam current density and beam intergration should be taken with care for the analysis of precious paper-like objects. There is a maximum

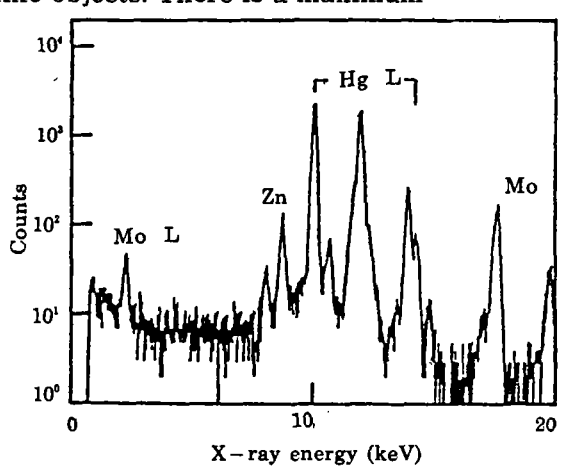

Fig.6 IXX spectrum of seal ink on one Chinese painting with a primary target of Mo: 2.6 MeV, 0.5 μ A, 300 s

limitation of beam integration for each type of paper under low beam current density,

above which considerable damage may occur to the paper samples. This paper deterioration is irreparable which hinders the accurate measurement of trace elements in paper－like samples and the repeated analysis.

For IXX analysis of paper－like samples, these limitations no longer exist. X－rays are relatively nondestructive and the analysis sensitivities can be improved by increasing the primary X－ray flux as high as possible. It is obvious that IXX is more suitable than PIXE for the analysis of documents, stamps, paintings and other paper－like samples which has attracted much attention in recent years[9－13]. Fig.6 shows a IXX spectrum obtained in the analysis of seal ink on a traditional Chinese painting, while a primary target of molybdenum and a proton beam current of 1 μ A were used.

Ⅳ. CONCLUSION

IXX technique is a useful method to solve problems encountered in PIXE analysis. It can be used to eliminate spectral interferences from major matrix elements of sample and significantly suppress the background by proper selecting the primary target. Because of its nondestructiveness, it is more suitable for the analysis of heatsensitive and delicate specimens especially the precious paper－like samples.

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