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**BORON NITRIDE PROTECTIVE COATING
OF BERYLLIUM WINDOW SURFACES**

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1. Introduction

The use of beryllium windows on white synchrotron radiation beamlines is constrained by the fact that the downstream surfaces of these windows should not be exposed to ambient atmosphere [1]. They should, rather, be protected by a tail-piece under vacuum or containing helium atmosphere. This tail-piece is typically capped by Kapton (3M Corporation, St. Paul, MN) or aluminum foil. The reason for such an arrangement is due to the health risk associated with contaminants (BeO) which form on the exposed beryllium window surfaces and due to possible loss of integrity of the windows [2]. Such a tail-piece may, however, add unwanted complications to the beamline in the form of vacuum pumps or helium supplies and their related monitoring systems. The Kapton windows may burn through in the case of high intensity beams and lower energy radiation may be absorbed in the case of aluminum foil windows. A more ideal situation would be to provide a coating for the exposed beryllium window surface, sealing it off from the atmosphere, thus preventing contamination and/or degradation of the window, and eliminating the need for helium or vacuum equipment.

2. Boron Nitride

As a result of a literature search for information concerning beryllium metal contamination [2], a U.S. Patent entitled **Protective and Lubricating Composition for Beryllium** was found [3]. This patent, granted in 1971, states the following:

ABSTRACT OF THE DISCLOSURE

A composition comprising a mixture of boron nitride powder and colloidal silica in a substantially volatile carrier is described. This material is coated on a beryllium surface and the volatile carrier evaporated to leave a dry film lubricant. The dry film lubricant is particularly useful for high temperature forming of beryllium and in addition provides oxidation protection thereby obviating need for a protective environment and minimizing safety hazards.

The patent details the formulation and uses of the boron nitride (BN) composition. It was evident that since the BN could protect beryllium metal during the forming process, it might also be useful in protecting beryllium windows on synchrotron beamlines. Loss of synchrotron beam flux would be minimal because the absorption edges of B and N are very similar to Be (Be K = 111.5 eV, B K = 188 eV, N K = 409.9 eV [4]).

A search for the components of the BN composition as described in the patent was undertaken. This resulted in finding that The Carborundum Company (Electronic Ceramics Division, P.O. Box 664, Niagara Falls, NY 14302) presently markets a product known as "Combat® Boron Nitride Aerosol Spray" in a 16 ounce (net wt.) aerosol container. The characteristics of this product as cited in their Technical Data sheet [5] are listed in Table 1. In addition, a methylene chloride carrier is used as well as a titanium binder. The presence of the Ti was determined by the author's analyses and confirmed by Carborundum (see Section 4.).

3. Application and removal of Boron Nitride aerosol spray

A certain amount of care must be taken when spraying the BN aerosol onto a Be window surface. The contents of the aerosol can are under high pressure and the spray plume is broad. Therefore, when spraying: a) mask all components around the window to prevent unwanted coverage of the surfaces by the BN; b) hold the spray can well away from the Be surface to prevent smearing of the BN as it lands on the Be surface; c) do not spray too thick a layer at any single time; d) it is beneficial to spray the window when it is already mounted in its assembly so the BN coats both the window and the joint

between the window and holder; e) spray in a well ventilated area (BN is classified as a "nuisance dust" and methylene chloride as a cancer hazard [6]). The author typically sprays to a visual uniform coverage of the window in question. Experience has shown that full spray cans produce a more uniform coverage than nearly empty cans. Removal of the aerosol coating from a Be window is best accomplished by wiping the surface with alcohol using a cotton swab or soft tissue.

4. Analysis and use of Boron Nitride aerosol spray

Prior to use on a beamline, the BN aerosol was analyzed to determine the nature of the binding agent (a proprietary formulation developed by a subcontractor manufacturing the aerosol using BN supplied by Carborundum). Both energy dispersive x-ray spectroscopy (EDS) and electron energy loss spectroscopy (EELS) revealed the presence of titanium [7]. The molecular form may be TiO_2 , but this is not certain. Carborundum confirmed this by their own emission spectrographic analysis which showed 0.4% Ti and 0.1-0.2% oxygen [8]. Ti may be an undesirable component due to its 4.966 keV k-absorption edge, a low energy region much in use at synchrotron radiation beamlines.

The BN aerosol has been voluntarily tested over the last 2.5 years on many National Synchrotron Light Source (NSLS) x-ray beamlines (X2B, X3A, X7A, X15A, X17B1, X17B2, X17C, X25, X27A+B+C) with a broad range of configurations [9]. In each case, the window sprayed was at least the second Be window downstream along the beamline, never the first. This assured maintenance of beamline vacuum integrity in the event the second Be window developed a leak. The coating has been very successful in preventing the accumulation of contaminants on window surfaces, thus greatly reducing window degradation. This is especially noteworthy on the X17 superconducting wiggler beamlines which have operated up to a critical energy (E_c) of 20 keV (standard NSLS x-ray bending magnet $E_c = 5$ keV) and on the X25 wiggler beamline which has achieved >100 wats/mm² power densities at the focal point. Of additional interest is the fact that at high power densities, the nitrogen in the BN fluoresces and clearly highlights the footprint of the beam as it passes through the Be window [10]. The majority of the participating beamlines report no beam degradation due to the presence of the BN on their windows.

5. Beamline configurations in which Boron Nitride is not recommended

Two beamlines independently reported similar beam perturbations due to the effect of BN coatings on their Be windows. The X2B microtomography beamline [11] uses a 2-D detector mounted downstream of a monochromator. At 10 keV, adjacent pixels (variable from 1.5-6.0 μ m) on the detector exhibited 30-50% differences in beam intensity when a BN coated window was located upstream of the monochromator. The detector exhibited uniform beam intensity when an uncoated Be window was used.

A similar condition was found on the X25 hybrid wiggler beamline [12]. A detector was mounted in-line with a 6 μ m pinhole and scanned in 10 μ m steps across an 8 keV beam transmitted through a BN coated Be window. Beam intensity varied up to 25% from point to point. This variation was eliminated when an uncoated window was installed (Fig. 1,2)[13]. Note that both of the above conditions arose due to the use of small pixel or pinhole sizes in which uniform beam transmission across a window was an experimental requisite.

The general opinion of the researchers was that the large variation in beam intensity could not be due to the BN and that the Ti binder played a more important role than previously expected [12,13]. Calculations showed that a 10 μ m thick piece of TiO_2 could cause a 30% reduction in beam intensity at 8 keV [14]. A fluorescence scan of a coated window sample conducted on the X26C beamline clearly demonstrated the presence of Ti in the BN (Fig. 3,4). A "region of interest" scan (10 μ m steps over 0.5

mm @ 5 sec/step) on the same window sample demonstrated >140% variations in Ti concentration over 10 μm distances (Fig. 5)[14]. These results are similar in nature to those on X2B and X25. A further confirmation of the Ti variation across the same coated window analyzed on X26C is shown in an EDS Ti x-ray dot map (Fig. 6)[7]. The scanning electron micrograph (Fig. 7) of the same area shows distinct gaps in the BN coating which correspond to the absence of Ti in the dot map [7].

The above results indicate that the variation in transmitted beam intensity across a BN coated window is due to a lack of uniformity in the sprayed coating. The degree of variation is due to differences in Ti concentration per unit area which is also a function of coating thickness. These findings demonstrate an extremely important point; the decision whether or not to use BN coatings on Be windows should be based, in part, on a) the need for uniform beam transmission at lower x-ray energies across the window, and b) the use of small pinholes or detector pixel sizes.

6. The future

Alternate coating methods have been proposed such as direct application of BN (without Ti) via chemical vapor deposition. The recent advances in application of diamond films also bear examination. The disadvantage of these two techniques is the loss of simplicity afforded by the aerosol spray method. Work by the author will continue in this field.

7. Acknowledgements

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The author would very much appreciate learning about anyone else's experiences with boron nitride aerosol coatings in the synchrotron radiation field.

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- [13] Brian Stephenson, IBM T.J. Watson Research Center, and Mark Sutton, McGill University; experimental results produced on X25 Beamline.
- [14] Mark Rivers, University of Chicago, X26C; absorption calculations and BN analyses for Ti.

9. Tables

Table 1: Properties of Combat® Boron Nitride Aerosol

Chemical formula	BN
Molecular weight	24.82
Specific gravity	2.25
Color	White
Crystal structure	Hexagonal
Particle size	<1 micron
Working temperature:	
a) Inert atmosphere	5400°F
b) Oxidizing atmosphere	1300°F
Dielectric strength	790 volts/mil
Electrical resistivity	$>2 \times 10^{14}$ ohms
Coefficient of friction	0.2-0.7

10. Figure captions and figures

Fig. 1. Detector scan across 8 keV beam transmitted through boron nitride coated beryllium window. Detector mounted in-line with 6 μm pinhole, scanning in 10 μm increments [12].

Fig. 2. Same conditions as Fig. 1 except beryllium window is uncoated [12].

Fig. 3. Fluorescence analysis (300 sec) of single area on BN coated portion of Be window [14].

Fig. 4. Fluorescence analysis (300 sec) of single area on uncoated portion of same Be window as Fig. 3 [14].

Fig. 5. Fluorescence scan for Ti (10 μm steps over 0.5 mm @ 5 sec/step) on same BN coated Be window as Figs. 3,4 [14].

Fig. 6. Energy dispersive x-ray spectroscopy (EDS) image showing x-ray dot map of Ti on same BN coated Be window as Figs. 3,5. Bar = 14 μm [7].

Fig. 7. Scanning electron micrograph (SEM) of same area shown in Fig. 6. Bar = 14 μm [7].

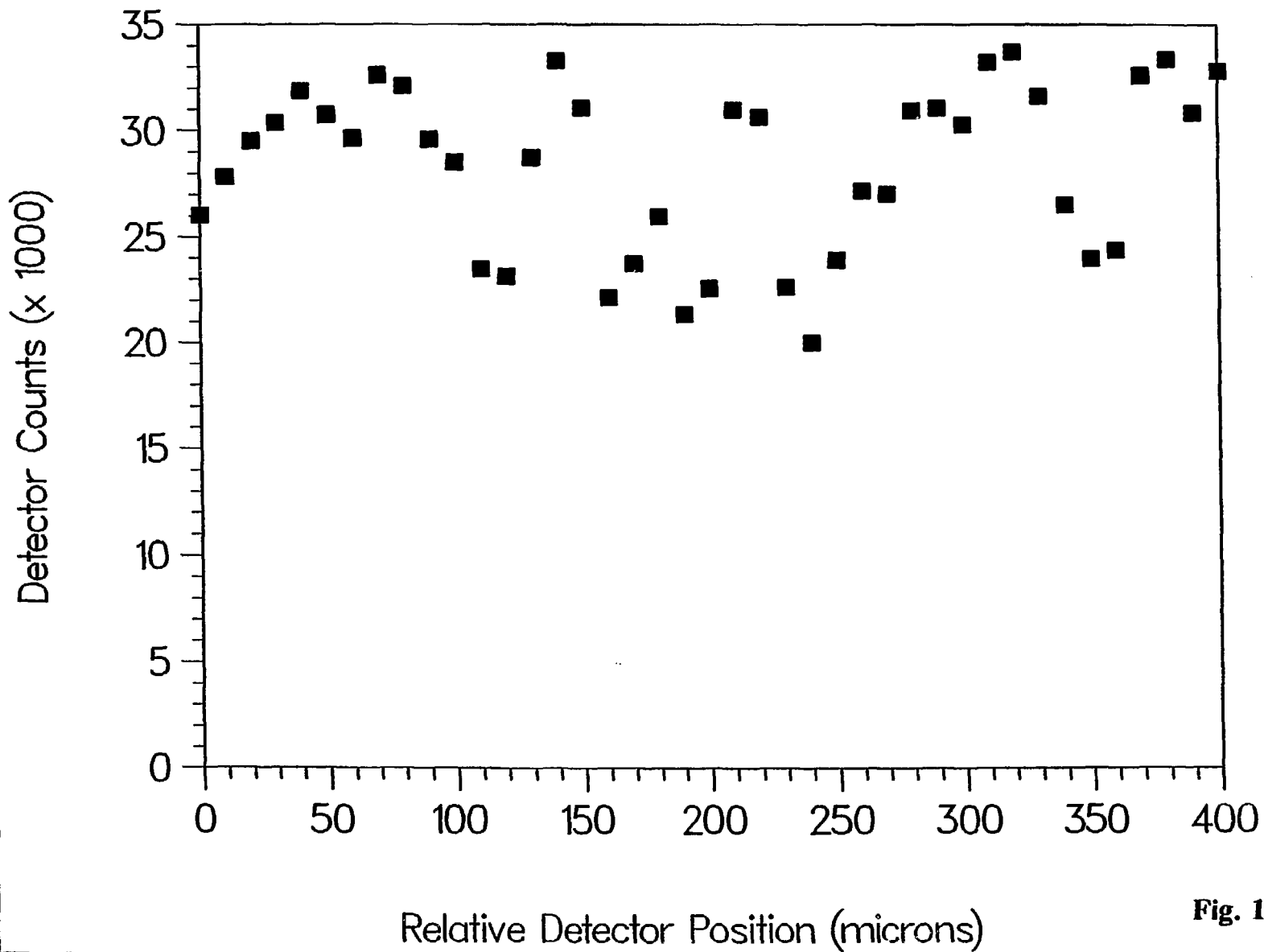


Fig. 1.

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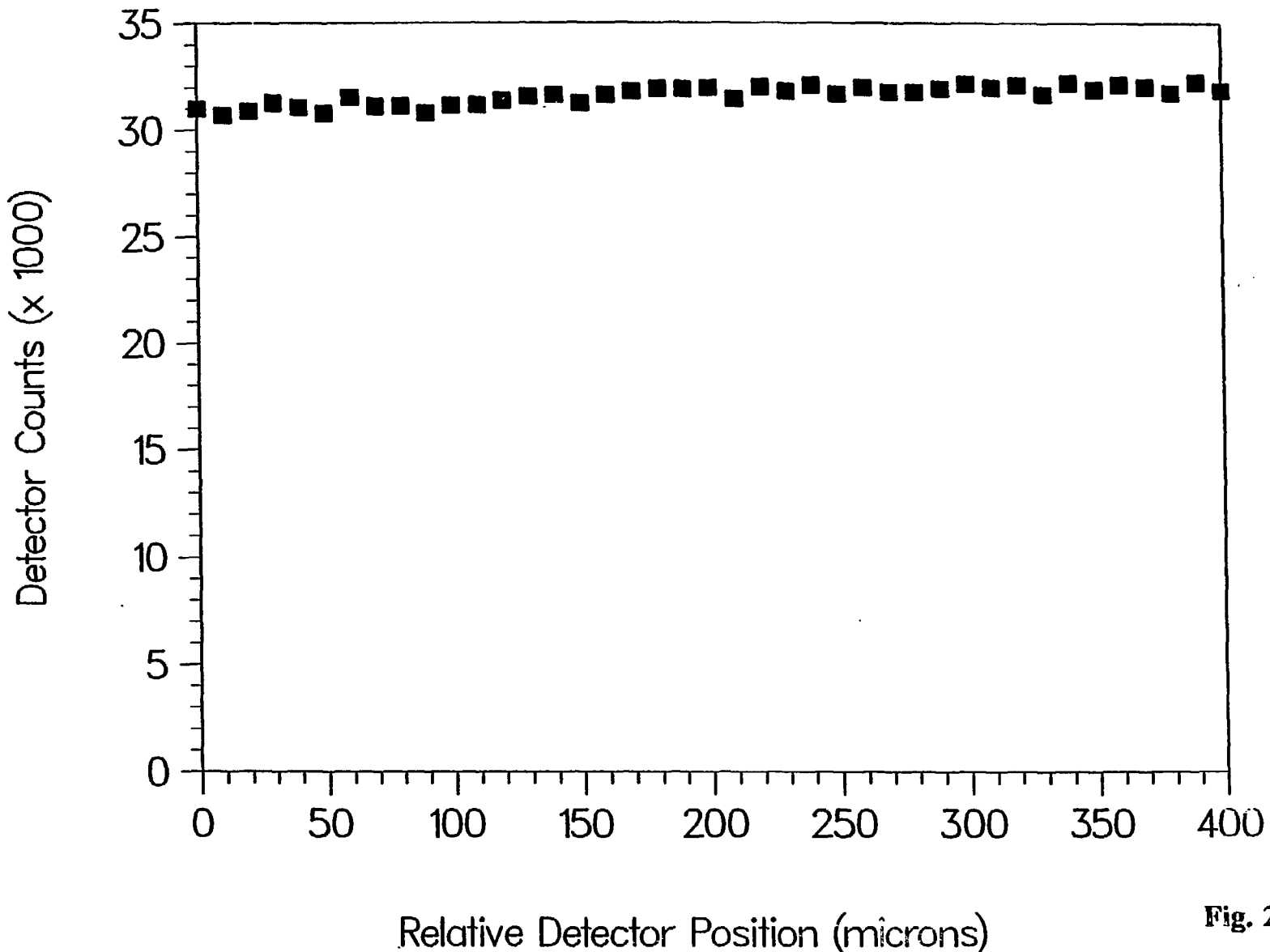


Fig. 2.

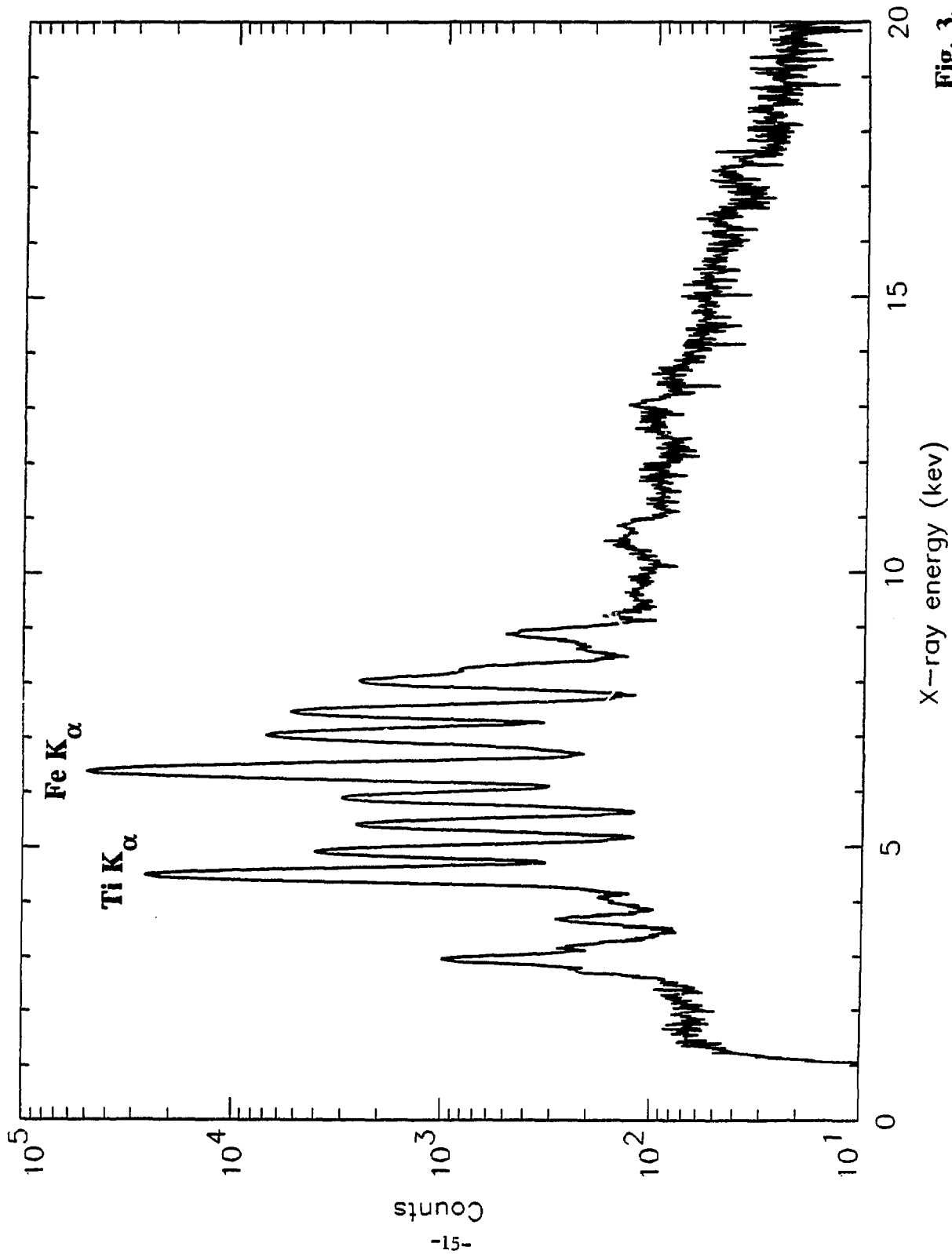


Fig. 3.

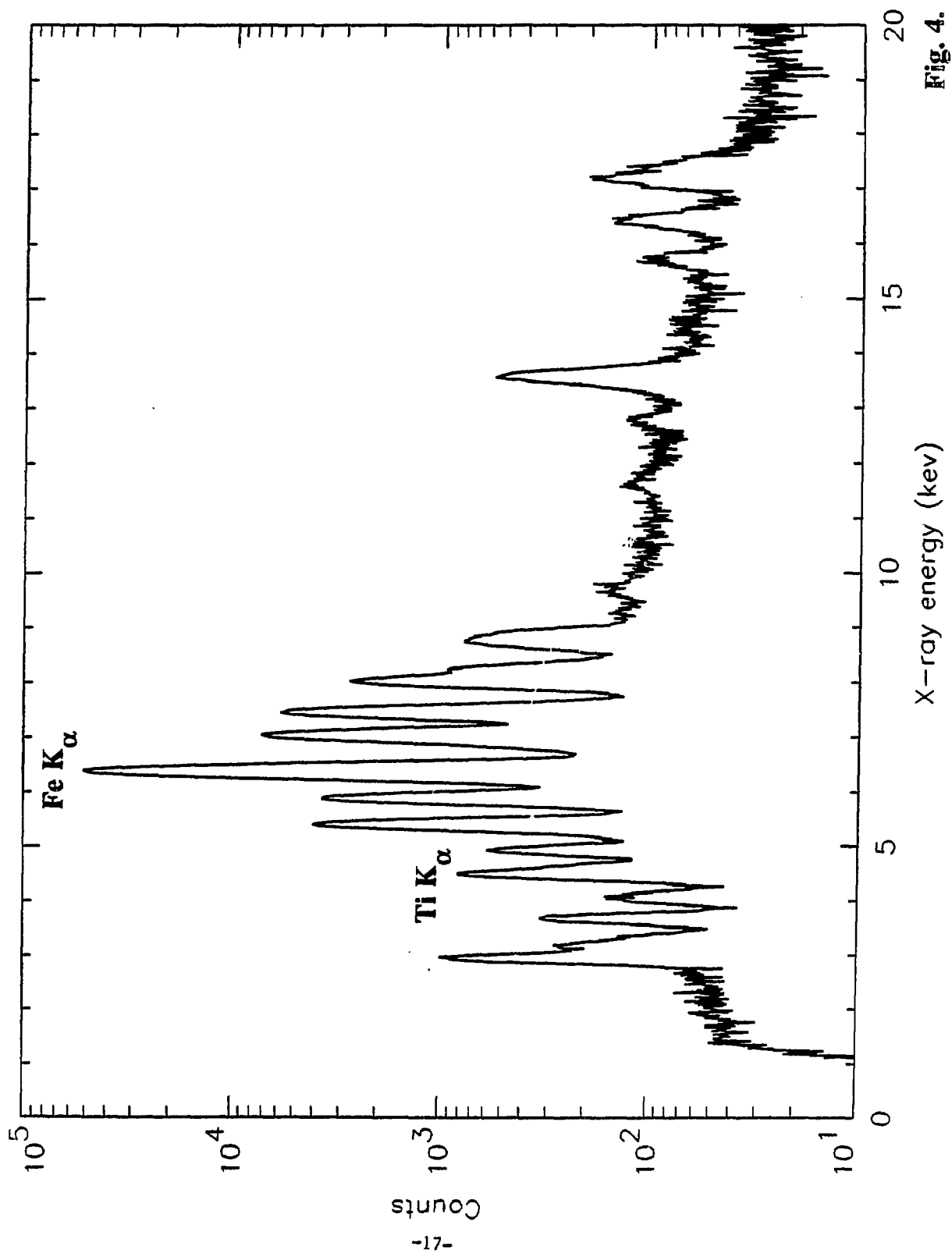


Fig. 4.

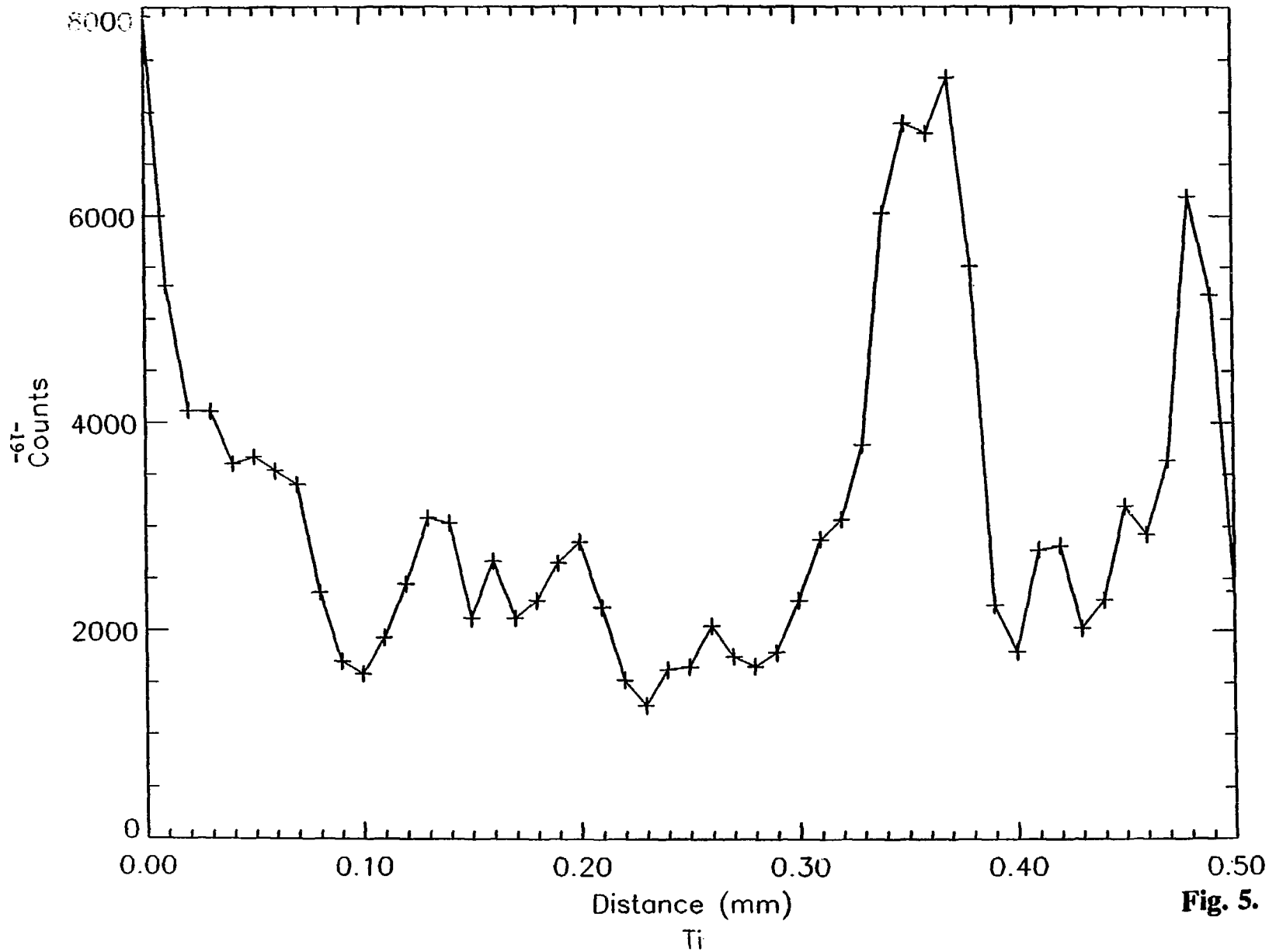


Fig. 5.

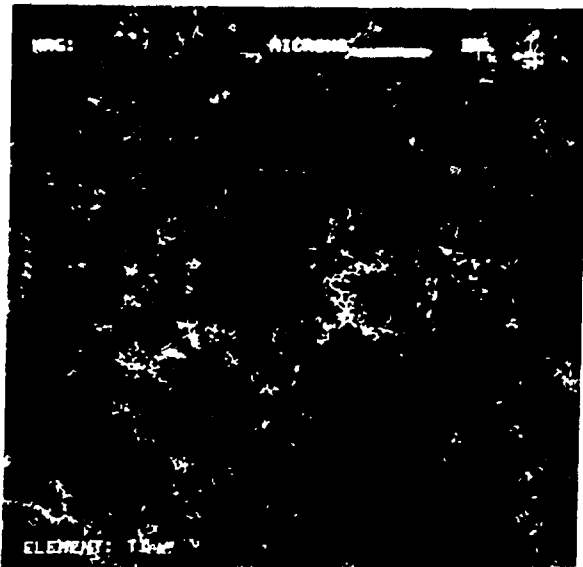


Fig. 6.

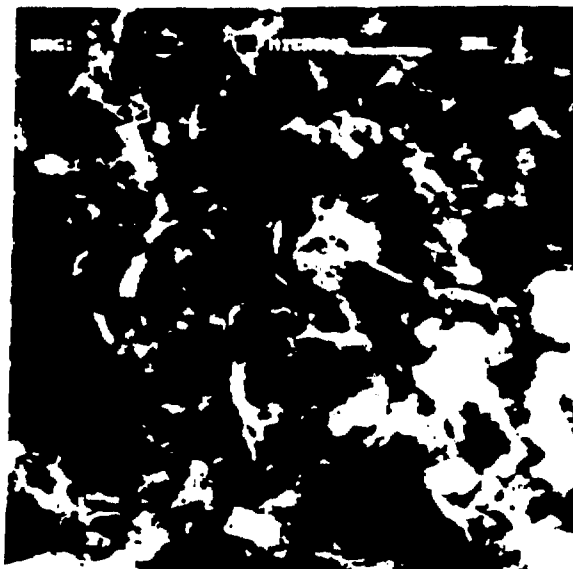


Fig. 7.