

POSITRON MICROANALYSIS WITH HIGH INTENSITY BEAMS DE91 005014

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INTRODUCTION

One of the more common applications for a high intensity slow positron facility will be the microanalysis of solid materials. In the first section of this paper some examples are given of procedures that can be developed. Since most of the attendees of this workshop are experts in positron spectroscopy, comprehensive descriptions will be omitted. With the exception of positron emission microscopy, most of the procedures will be based on those already in common use with broad beams. The utility of the methods have all been demonstrated, but material scientists use very few of them because positron microbeams are not generally available. A high intensity positron facility will make microbeams easier to obtain and partially alleviate this situation.

All microanalysis techniques listed below will have a common requirement, which is the ability to locate the microscopic detail or area of interest and to focus the positron beam exclusively on it. The last section of this paper is a suggestion of how a high intensity positron facility might be designed so as to have this capability built in. The method will involve locating the specimen by scanning it with the microbeam of positrons and inducing a secondary electron image that will immediately reveal whether or not the positron beam is striking the proper portion of the specimen. This 'scanning positron microscope' will be a somewhat prosaic analog of the conventional SEM. It will, however, be an indispensable utility that will enhance the practicality of positron microanalysis techniques.

MICROANALYSIS METHODS USING INTENSE POSITRON BEAMS

Positron Emission Microscopy

Brandes et al. (1) have demonstrated that positron emission microscopy can be performed at high resolutions. The microbeam achieved in a SPM facility will make such a technique semi-routine. The positron emission microscopy method involves the projection and magnification of positrons emitted from a small area of a moderator. The authors (2) have described how resolution and geometrical magnification is limited by the signal-to-noise of the microchannel plate on which the image is recorded. Typical noise levels of microchannel plates are 1 count $\text{sec}^{-1} \text{cm}^2$. To record an intelligible image, the positron intensity should be at least 4 times the noise level. The positron intensity projected from the remoderated spot will be inversely proportional to the square of the geometrical magnification of the microscope. In order to achieve a geometrical magnification of 1 million, which should yield a resolution of about 2.5 nm, the intensity of positrons

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emerging from the remoderated spot must be greater than about $4 \times 10^{12} \text{ sec}^{-1} \text{ cm}^{-2}$. The slow positron fluxes expected from the HFIR and ANS facilities will be greater than this. Resolutions of positron emission microscopes will reach their full capabilities, and will be limited only by the aberrations of the objective lenses.

Positron Induced Auger Electron Spectroscopy

Weiss et al (4) have demonstrated that this technique has a unique sensitivity for the uppermost atomic layers of solid surfaces. Also, signal to noise levels are significantly lower. This technique applied on a microscopic basis will have a strong impact on surface analysis.

Angular Correlation of Annihilation Radiation (ACAR)

Intense positron beams will make it possible to perform ACAR measurements on microscopic specimens. Such measurements will be useful for determining the electron momentum distributions and defect concentrations of specimens such as the individual grains in polycrystalline specimens. By varying the positron energies, the depth of penetration can be made very small, allowing comparison of the grain boundary areas, on the surfaces of the grains, with the interiors of the grains.

Doppler Broadening of Annihilation Radiation

Doppler broadening spectroscopy will most likely be the most widely used technique for positron microanalysis. The Doppler broadening information will be similar to that of ACAR; the information will not be as detailed, but collection times will be faster. Its capability for revealing the presence of vacancies and other defects has been demonstrated many times for large specimens. Sharma et al. (4) have shown that Doppler broadening measurements can reveal defect distributions around cracks and strained regions of solid specimens.

Positron Lifetime Spectroscopy

The positron microbeam can be bunched so as to provide start signals in positron lifetime measurements. With this capability, all of the many applications of lifetime spectroscopy, such as studies of defects and free volumes, can be directed toward microscopic specimens.

Positron Channeling

Positron channeling studies, such as those of Schultz et al. (5) are being done on larger specimens. The SPM would make these possible on smaller specimens. Channeled positrons travel exclusively between rows of atoms in crystals. Measurements of x-ray fluorescence intensities of impurity elements as a function of channel conditions will allow the determination of whether impurity elements are substitutional or interstitial. Interstitial

impurities will have higher fluorescence intensities when the positrons are channeled, whereas the substitutional impurities will have higher intensities for dechanneled conditions.

Microelectronics Device Technology

One of the most obvious avenues of application for positron microanalysis is in the characterization of microcircuitry and electronic devices. Work such as that of Tanigawa et al. (6) on semiconductor junctions will be performed on microcircuits.

THE NEED FOR A SCANNING POSITRON MICROSCOPE DEVICE

Given a sufficiently high current of slow positrons, a "SPM" (scanning positron microscope) can be designed that will operate in a mode that is essentially the same as that of the conventional scanning electron microscope (SEM). The location of microscopic specimens in the SEM is done without great difficulty. Specimens as small as 1 micrometer in dimension are located by means of secondary electron images. By reducing the scan raster to successively smaller dimensions, higher magnification images are generated, and the electron beam becomes exclusively focused on the microspecimen. It should be possible to utilize a scanning positron microscope in a similar fashion. It might be assumed that the incorporation of an optical microscope in the positron facility will be adequate for locating the microspecimen and focusing the beam. The authors have had extensive experience in conventional scanning electron microscopy; they have found that the technique of imaging the microspecimen by means of the electron beam provides much better assurance that the electron beam is focused on the proper area, rather than some region nearby. Consider the common problem of the elemental analysis of a small particle by means of x-ray fluorescence induced by the electron beam: by generating and observing the secondary electron image of the particle during the time that the x-ray fluorescence spectrum is collected, one has assurance that the beam has not strayed from its intended target. An optical microscope is not indicated for the "SPM" shown in Figure 1, but no doubt it would be useful. The magnification of the optical microscope will be limited to 50 X or lower, however, because the working distance between the specimen and the objective lens will necessarily have to be large to accommodate the other equipment.

Figure 1 is a schematic diagram of how a scanning positron microscope might be constructed. The positron beam will be brightness enhanced and scanned across the specimen. The energy of the positron beam will be variable in the range of 1-20 keV. The secondary electrons generated will be collected by a channel electron multiplier and recorded in a single-particle counting mode. A cathode ray tube whose scan raster is synchronized with the positron beam scan will fire each time a secondary electron is recorded. In this manner a secondary electron emission map of the specimen will be generated. The resolution that will be achieved

for the image will depend on the brightness of the positron beam.

When a positron beam strikes a solid specimen, secondary electrons are emitted with an efficiency that is about the same as that for electron bombardment. If a sufficiently high current of positrons were available, the SPM could achieve resolutions as high as those of the SEM. Typical electron currents in SEM's are over one nanoampere. For the positron facilities that will be available during the next 10 years, we can expect at most 0.01 - 0.1 nanoampere after brightness enhancement. Nevertheless, this will be enough for low-magnification imaging. High resolution SEM's, operating in the secondary electron imaging mode, can achieve resolutions as small as 5 nm. With the SPM, the resolution will probably be no better than 5000-10,000 nm, but this will be sufficient to allow many new types of positron microanalyses to be done. The purpose of the SPM will not be high resolution imaging. Its main function will be to allow the microspecimens to be located. After the specimen is located and the positron beam is injected into it, the effective resolution will be no better than 100 nm, no matter how small the positron beam cross section. When positrons or electrons are injected in solids the beams are dispersed over volumes that are typically of this dimension. For most positron microanalyses the resolved specimen will be at least 0.1-1.0 micrometer in dimension.

The electron multiplier indicated in Figure 1 will detect electrons in a digital mode. It is anticipated that the available positron current might be too low to generate enough secondary electrons for detection by the scintillator devices normally used in scanning electron microscopes. It may be necessary to generate the secondary electron image by single particle counting. The signal-to-noise will be poor for such an image, but the resolution should still be adequate to locate and isolate the micron-sized specimens on which the spectroscopy is to be performed.

A Si(Li) detector for recording x-ray fluorescence induced from the specimen by the positron beam is also indicated in Figure 1. This will serve as a convenient method of specimen identification and location also. However, there will probably be many applications in which it will be necessary to identify elements in the specimen for which the positron spectroscopy is to be performed. For example, if an alloy is being studied, it may be desirable to measure defect content of the different grains as a function of elemental composition. The cross section for generating core holes in atoms, which lead to x-ray fluorescence, is not as high for positrons as for electrons, but the method will be practical nevertheless.

CONCLUSIONS

Given a sufficiently high beam intensity, positron microanalysis will become a practical supplement to the more conventional electron beam, ion beam, and x-ray methods. Positron microanalysis will provide information that is otherwise unavailable.

All microanalysis problems will have the common requirement of being able to locate the microspecimen and to focus the positron beam exclusively on it. The most practical way of accomplishing this is by means of a scanning positron microscope (SPM) built into the high intensity slow positron facility. The resolution of the SPM will not rival that of the conventional SEM, but it will serve as a general utility for all of the microanalysis problems.

REFERENCES

1. G. R. Brandes, K. F. Canter, A. P. Mills, Jr., Phys. Rev. Lett. 61, 492 (1988)
2. L. D. Hulett, Jr., J. M. Dale, S. Pendyala, Materials Science Forum 2, p 133, (Trans. Tech. Publications Ltd., Switzerland, 1984)
3. A. Weiss, R. Mayer, M. Jibaly, C. Lei, D. Mehl, K. G. Lynn Phys. Rev. Lett., 61, 2245-48 (1988)
4. S. Sharma, R. M. Johnson, Y. J. Ataiyan, L. M. Diana, P. J. Coleman, Positron Annihilation, p 467, North Holland, New York, (1982) 1. Pendyala, secondary electron emission.
5. P. J. Schultz, L. R. Logan, T. E. Jackman, J. A. Davies, Phys. Rev. B, 38, 6369 (1988)
6. S. Tanigawa, Proceedings of Third Conf. on Positron and Positronium Chemistry, July 1990, Marquette University, Milwaukee, WI, e.d. by D. Schrader, Y. C. Jean, World Scientific.

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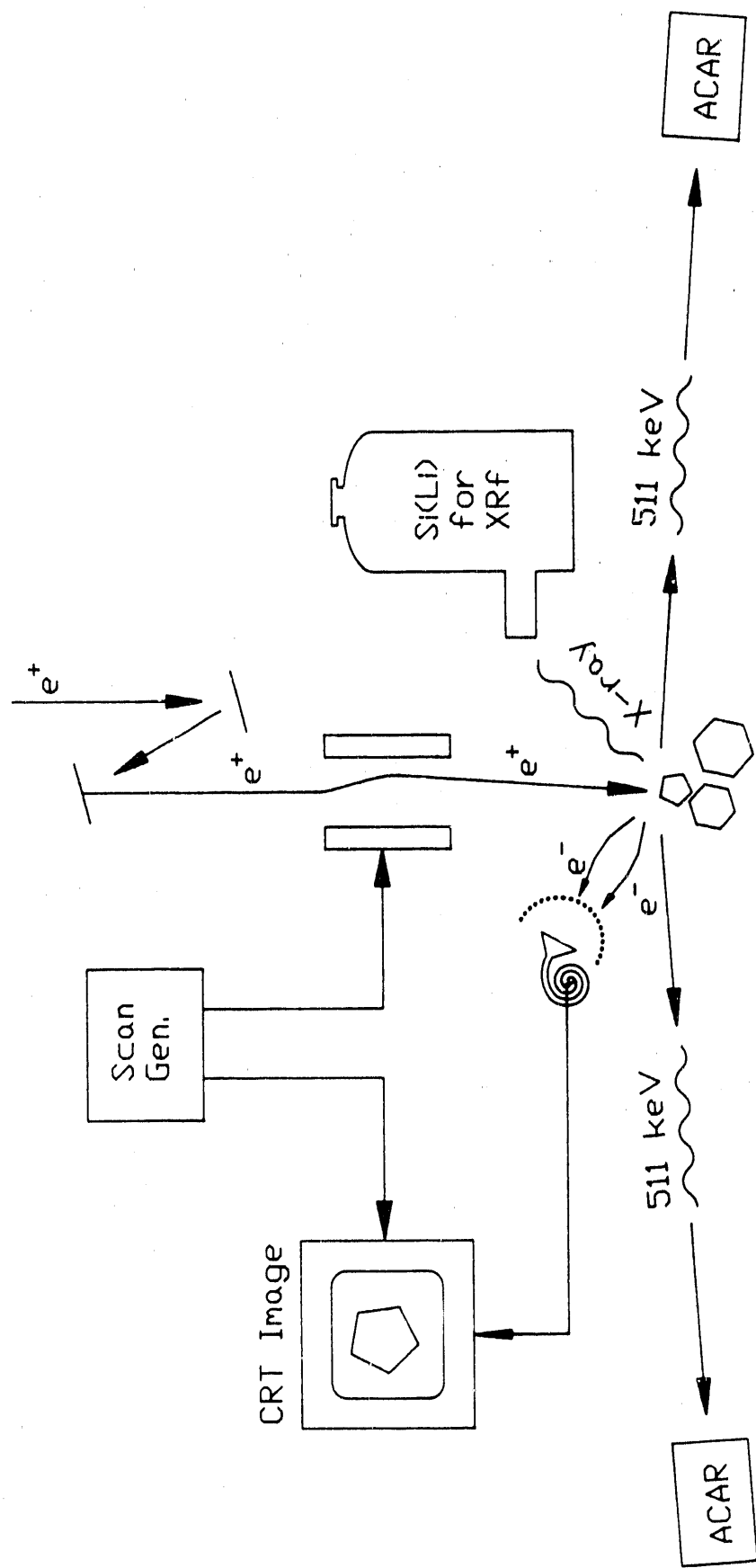


Figure 1. Schematic diagram of scanning positron microscope, for use in the isolation of microscopic specimens on which positron spectroscopy measurements are to be made.

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