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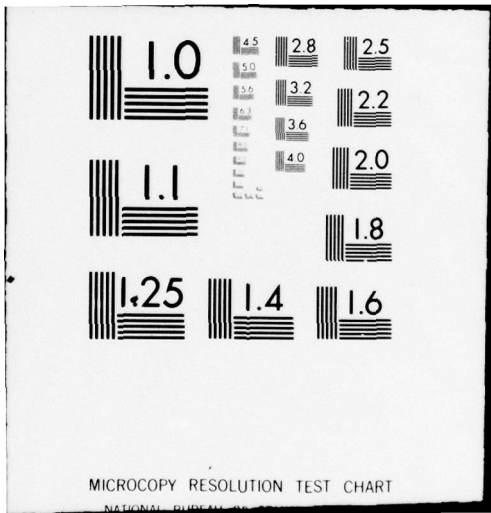
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**CHARACTERIZATION OF LPE GROWN HgCdTe
BY ION SCATTERING SPECTROMETRY.**

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W. L. BAUN

MECHANICS AND SURFACE INTERACTIONS BRANCH
NONMETALLIC MATERIALS DIVISION

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Final Report for period July 1978 - July 1979

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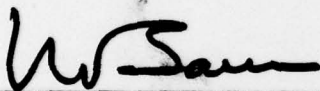
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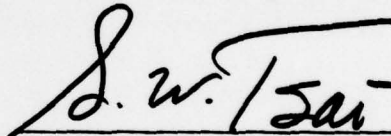
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Results are shown here for low energy ion scattering spec- trometry (ISS) experiments on mercury cadmium telluride (HCT) grown by liquid phase epitaxy (LPE) on CdTe substrates. By proper etching of the substrates and control of epitaxial growth conditions, surfaces of the zinc blend structure may be obtained in which {111} faces have a top layer of cadmium and mercury (A sites) or of tellurium (B sites). Ion scattering experiments at low energies (500 V or less) on epitaxial HCT show only A or		

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B sites. However, when the helium or neon ion beam potential is raised to values conventionally used (1500-2500 V), then energy losses from both A and B atoms are observed. At these higher energies the ions sputter or displace the outer atoms or tunnel through to interact with atoms in the second layer. Spectra obtained at these higher energies show interesting fine structure which suggest that energy losses are occurring in multiple collisions and nonbinary scattering. Scattered ion yield curves, which for some elements show oscillatory fine structure, are dramatically different for A and B surfaces. Even though these yield curves are very complicated because of the structure and number of elements, the fine structure may prove useful for further understanding of interaction of neighboring atoms. Yield curves along with low energy ion scattering spectra provide a unique method of analysis of the outermost atomic layer of epitaxial films.

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FOREWORD

This technical report was prepared by W. L. Baun, Mechanics and Surface Interactions Branch, Nonmetallic Materials Division, Air Force Materials Laboratory (AFML/MBM). This work was initiated under Project 2303 "Chemistry Research," Task 2303Q1 "Surface Chemistry" and W. U. D. #41 "Analysis of Traveling Wave Tubes." The Work Unit Monitor is Capt. Bruce Lamartine.

This work was done inhouse preparatory to studying traveling wave tube cathode activation phenomena to determine sensitivity of ISS to composition of the first atomic layer on the surface. This model system was studied during the period November 1978 and July 1979.

The author wishes to thank Ross P. Murosaka for supplying the samples of epitaxially grown HgCdTe on CdTe. Technical discussions with T. W. Haas have proven useful in this work.

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FIGURE

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10 Repetitive Scans of the $^4\text{He}^+$ Ion Scattering,
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SECTION I
INTRODUCTION

Epitaxial growth of films has actually been studied over only the last 30 years, except for a few early studies, but evidence of such growth in nature has existed for many, many years.¹ Numerous materials have been grown on a whole host of substrates by several methods such as vacuum evaporation (to include molecular beam epitaxy, MBE),² electrodeposition,³ chemical vapor deposition (CVD),⁴ sputtering,⁵ and liquid phase epitaxy (LPE).⁶ The process of forming epitaxial films has technological importance in several fields, notably the semiconductor industry where III-V, II-VI, and IV-VI compounds grown by the above methods are used for a variety of purposes. Several methods have been used to characterize the surface of epitaxial films. Transmission electron microscopy (TEM) and scanning electron microscopy (SEM) give information on morphology and thickness of films. Low energy electron diffraction (LEED) is extremely sensitive to structure and is often teamed with Auger electron spectroscopy (AES) which determines the distribution and concentration of elements at the surface. The AES method is a very sensitive tool for detection of impurities and determination of stoichiometry of thin films. It does, however, have a finite sampling depth due to the penetration of electrons (2-4 KeV energy) and the variable depth from which the secondary Auger electrons can escape from the bulk. AES then provides an averaged analysis over this finite sampling volume.

It is possible in some binary and ternary semiconductor systems to selectively etch either the substrates or epitaxially grown films to expose at the surface only atoms of one species, so called "A" or "B" surfaces.⁷ Etching characteristics and resulting morphology are frequently used to identify such

surfaces since techniques using an electron beam (such as AES) or an x-ray beam (such as photoelectron spectroscopy) provide the average analysis discussed above.

A technique which does not suffer the disadvantage of averaging over a finite depth is ion scattering spectrometry (ISS).^{8,9,10} In this method a low energy ion beam interacts only with the outermost surface atoms to provide a true surface analysis. In addition to giving elemental distribution, fine features in the spectra can often be used to infer structural information.^{11,12,13} It is the purpose of this report to show results of ISS experiments on LPE grown HgCdTe on CdTe and to determine the experimental conditions necessary to achieve sampling from only the outermost surface layer.

1. EXPERIMENTAL

Surfaces were characterized by ion scattering and secondary ion mass spectrometries. Ion scattering spectrometry is performed by bombarding a surface with noble gas ions and measuring the energy distribution of the backscattered ions which will show energy losses predicted by a simple elastic collision theory. The elemental distribution of the first atomic layer may be obtained by scanning the energy ratio E/E_0 , where E_0 is the original energy of the ion and E is the energy of the rebounding ion after encountering the surface. Since the same beam which scatters from the surface also transfers energy to surface atoms, causing sputtering, continued sampling of the surface can result in an elemental profile with depth. The equipment used here and shown schematically in Figure 1 is a commercial unit (3M Co., St. Paul, MN) using a cylindrical mirror analyzer (CMA) with co-axial ion gun which permits acceptance of ions scattered at 137.7° . The ion gun produces a focused beam which will resolve about 100 micrometers and which may be rastered on the specimen and the signal gated to allow elemental images of the surface to be obtained.

The ISS experiments are performed in an ultrahigh vacuum chamber which is pumped by ion pumps and titanium sublimation pumps. An LN₂ cryo panel is also available in the system. The system is customarily pumped to about 5×10^{-9} Torr, valved off from ion pumps, fresh titanium evaporated from the sublimator, and the chamber backfilled to about $2-3 \times 10^{-5}$ Torr. The ion source is then activated, an acceleration potential between about 300 and 5000 V is selected, and ion beam parameters such as size and shape are adjusted. Samples were prepared by the Defense Electronics Division of Honeywell, Inc., under Air Force Contract F33615-77-C-5156. The HgCdTe was grown using the LPE method on CdTe substrates oriented to have {111} faces.

SECTION II
RESULTS AND DISCUSSION

1. CADMIUM TELLURIDE SUBSTRATES

CdTe {111} was used as a substrate for liquid phase epitaxial growth of HgCdTe. CdTe is one of the groups of II-VI semiconductors conventionally referred to as $A^{II} B^{VI}$ compounds. It crystallizes in the structure shown in Figure 2 which is a simple derivative of the diamond structure and also typical of the many III-V semiconductors. Since each Group II atom is tetrahedrally surrounded by Group VI atoms and vice versa, it is reasonable to assume that on the average each atom has four valence electrons. This suggests that the bonding has a covalent character and that the semiconducting properties of these compounds are similar to those of the corresponding Group IV element. This is also similar to III-V semiconductors. In II-VI compounds covalence is also likely but the increased electronegativity of the Group VI element introduces considerable ionicity. The forbidden energy gap for CdTe is 1.45 eV which corresponds to photon energy in the visible wavelength region. Such crystals therefore are used extensively to detect and measure visible light radiation.

He⁺ ion scattering spectra, taken at the usual ion beam potential used for ISS (1500-2500 V) on {111} surfaces of CdTe which were used as substrates for the HgCdTe failed to produce sufficient resolution to separate Cd and Te. Spectra using ²⁰Ne gave partial peak separation as shown in Figure 3 for 1000 and 1500 V ions. As can be seen from the spectra taken at low ion beam energy (<500V), only Te produces a scattered peak in the spectrum. Many repetitive scans taken with increasing primary ion energy up to 2500 V are shown in Figure 4 and indicate that a linear increase in scattering is seen from Cd. In the {111} orientation of CdTe, the Te atoms lie just above the Cd atoms, and also the

Te atoms (2.22 Å) are much larger than the Cd atoms (0.97 Å). This geometrical arrangement appears to shield the Cd atoms from the impinging ion beam. At higher voltages the ions apparently are able to penetrate sufficiently to scatter from Cd. A second interpretation of the phenomenon is that the higher energy ions selectively sputter Te to expose Cd atoms. If this happens, a rapid diffusion process must restore the crystal to original stoichiometry since one can return to lower energy ions and observe only Te on the surfaces which have been bombarded with high energy ions. A third possible interaction which may also explain unusual behavior in the epitaxial films is channeling of ions along certain directions to allow scattering from underlying atoms. Another interesting and related feature in the ion scattering spectrum using $^{20}\text{Ne}^+$ is the apparent shift to lower energy of $^{20}\text{Ne}^+$ scattered from Cd with respect to the Te peak. The "Cd peak" appears at lower energy than predicted by the simple binary collision equation. This may suggest that the ion interacts with Te as it passes through the first layer before or after scattering from Cd. This phenomenon is similar to one observed for tantalum in tantalum oxide but the exact mechanism of the energy loss is not clear.¹⁴ Regardless of the mechanism involved in these interactions, this shadowing of the Cd by Te illustrates the high surface sensitivity of ISS as compared with other methods of surface analysis such as AES or XPS where the signal is averaged over a finite depth.

2. EPITAXIAL HgCdTe

The HgCdTe was grown by the LPE method on the CdTe substrates prepared to result in {111} A surfaces having exposed A atoms (Cd and Hg) or {111} B surfaces having exposed B atoms (Te). The B surfaces scattered all ions (He, Ne, Ar) as expected showing only Te atoms on the surface. The A surfaces showed interesting scattering effects which suggest complex ion-atom interaction.

Figure 5 shows changes observed in the ${}^4\text{He}^+$ spectrum at two different ion beam potentials. With low energy ions (400 V) probably only scattering from Cd and Hg are observed. However, Cd and Te are not resolved with ${}^4\text{He}^+$, and it is possible that some contribution exists from Te. At higher energies such as shown for 1500 V, the contribution from Te increases, changing relative scattering intensities and broadening the Cd-Te line. A comparison of ${}^4\text{He}^+$ and ${}^{20}\text{Ne}^+$ scattering from A and B is shown in Figure 6. The B surface interacts with the ${}^{20}\text{Ne}$ probe ion just as it did with ${}^4\text{He}$; only Te is observed. The A surface, however, shows unusual behavior. In this spectrum all three elements seem to be observed, but it is possible that the line marked Te in the A surface spectrum is not actually due to Te but from some other source. The spectra are drawn to scale here, showing Te in A surfaces to scatter stronger than the Te on the B surface. The spectra seem to be very dependent on orientation. Other samples cut from the same epitaxial layer but oriented randomly in the specimen holder showed much different scattering characteristics, with Te only observed at high ion beam potential. Such specimens were sampled in six areas to check for inhomogeneities. None were found. The present experimental apparatus has no provision for rotation or change of angle of incidence, so no experiments on orientation could be made. It was thought that perhaps the "Te line" in the A spectrum might represent Ne ions which lost energy during channeling through the lattice and not represent any interaction with Te atoms. However, an experiment using argon ions as shown in Figure 7 shows virtually identical results as with neon except that Hg scatters argon relatively more efficiently. The "Te line" again appears to have a definite relation to an interaction between Te and Ar^+ , but just what mechanism would result in a shift to higher energies is not known. The apparent increase in scattering efficiency of {111A} surfaces also is not understood.

SECTION III
SCATTERED ION YIELD

Measurements of the number, or yield, of singly scattered ions as a function of ion energy may be used for surface characterization. Simple classical scattering calculations suggest the yield is a combination of the elastic scattering cross-section, a monotonically decreasing function of primary energy, and the probability of the scattered ion remaining ionized (monotonically increases with ion velocity). The resultant curve would then appear to be a structureless, smoothly changing curve perhaps dominated by one or the other of the above functions. However, Erickson and Smith¹¹ and Rusch and Erickson¹² have recently observed structure in the energy dependence of scattered ion yields which indicates electronic interactions occur during scattering causing structure on the yield curve. In some cases, oscillatory structure is observed such as that shown for $^4\text{He}^+$ on Pb in Figure 8 from work of Rusch and Erickson.¹³ The heavy dashed line in this figure highlights the pronounced oscillation in the elastically scattered ion signal which occurs as a function of ion energy. Not all yield curves show such dramatic structure. Rusch and Erickson¹³ have classified yields from scattering of He^+ , Ne^+ , and Ar^+ into four categories based on curve shape. The scattered ion yield curves were obtained here by slowly increasing the primary ion beam energy while rapidly and repetitively scanning the scattered ion beam energy (spectrometer voltage). This method has the advantage of recording the entire spectrum compared to recording only the intensity at the scattered ion peak.

The differences in yield curves obtained from A and B surfaces are dramatic as seen in Figure 9 for $^3\text{He}^+$. Here the B surface spectra show the He^+ interaction with the Te atoms on

the surface produces oscillatory structure in the yield curve. The A surface is much different, with both scattered peaks giving smooth featureless curves. Note that the Ne^+ also present in the system when the B surface was recorded shows no structure on the scattered ion yield curve. It is also interesting that the largest peak in the A spectrum is probably an unresolved mixture of Cd and Te, as mentioned earlier, but it shows no apparent structure.

Rusch and Erickson¹³ have categorized the scattered yield of helium from Cd as Class I which is featureless and agrees with data from the A surface. They did not obtain data for Hg, but predictions based on electronic structure suggest that the curves would be similar to platinum and gold, which are also Class I, agreeing with that found for the A surface. Results for Te from the B surface did not agree with Rusch and Erickson's category IV curve. Their category IV shows some slowly varying structure, but not sharp oscillatory fine structure. When yield curves for elemental Te were recorded using He^+ a smooth curve perhaps showing subtle features was obtained, in agreement with Rusch and Erickson.

A comparison of results for $^4\text{He}^+$ scattering on Te from elemental Te and from the B surface is shown in Figure 10. Note that these spectra are from $^4\text{He}^+$ and the earlier yield curves were from $^3\text{He}^+$. Similar curves are obtained in both He isotopes but some energy shifts in the features are observed. It is obvious that the change in structure and bonding of Te in HgCdTe compared to elemental Te has allowed charge transfer between ion and atom. Oscillatory structure for certain elements is attributed to quasi-resonant charge transfer between vacant He^+ 1s states and the outer d states of these atoms. Normally, in the elemental form, the energy of the N_{IV} level of

Te (39.8 eV) would not provide an energy match for K electrons in He (24.6 eV). Perhaps the formation of molecular orbitals of d states provide a match between He^+ and Te to allow charge transfer. The formation of CdTe and HgCdTe results in a structure and packing similar to that of Sn. Sn has a 5d level (23.9 eV) which closely matches the He^+ 1s level and gives oscillatory yield structure very similar to that seen for Te in HgCdTe.¹⁵ If such correlations could be confirmed in other semiconductors, yield curve structure could perhaps be used as a tool to study bonding and formation of molecular orbitals.

SECTION IV
CONCLUSIONS

Ion scattering spectrometry may be used to characterize compound semiconductors such as CdTe and to study epitaxial growth of HgCdTe. The technique is sensitive to the outermost atomic layer. Fine features in the spectra provide insights into structure and the use of yield curve features may allow prediction of bonding characteristics. This work establishes that sensitivity to only the first atomic layer may be obtained with ion scattering when ion beam energy is kept low, in the neighborhood of several hundred volts. The numerous spectral fine features which occur perhaps raise more questions than provide answers, but nevertheless they provide the exciting possibility of using ISS to determine structure and chemical binding.

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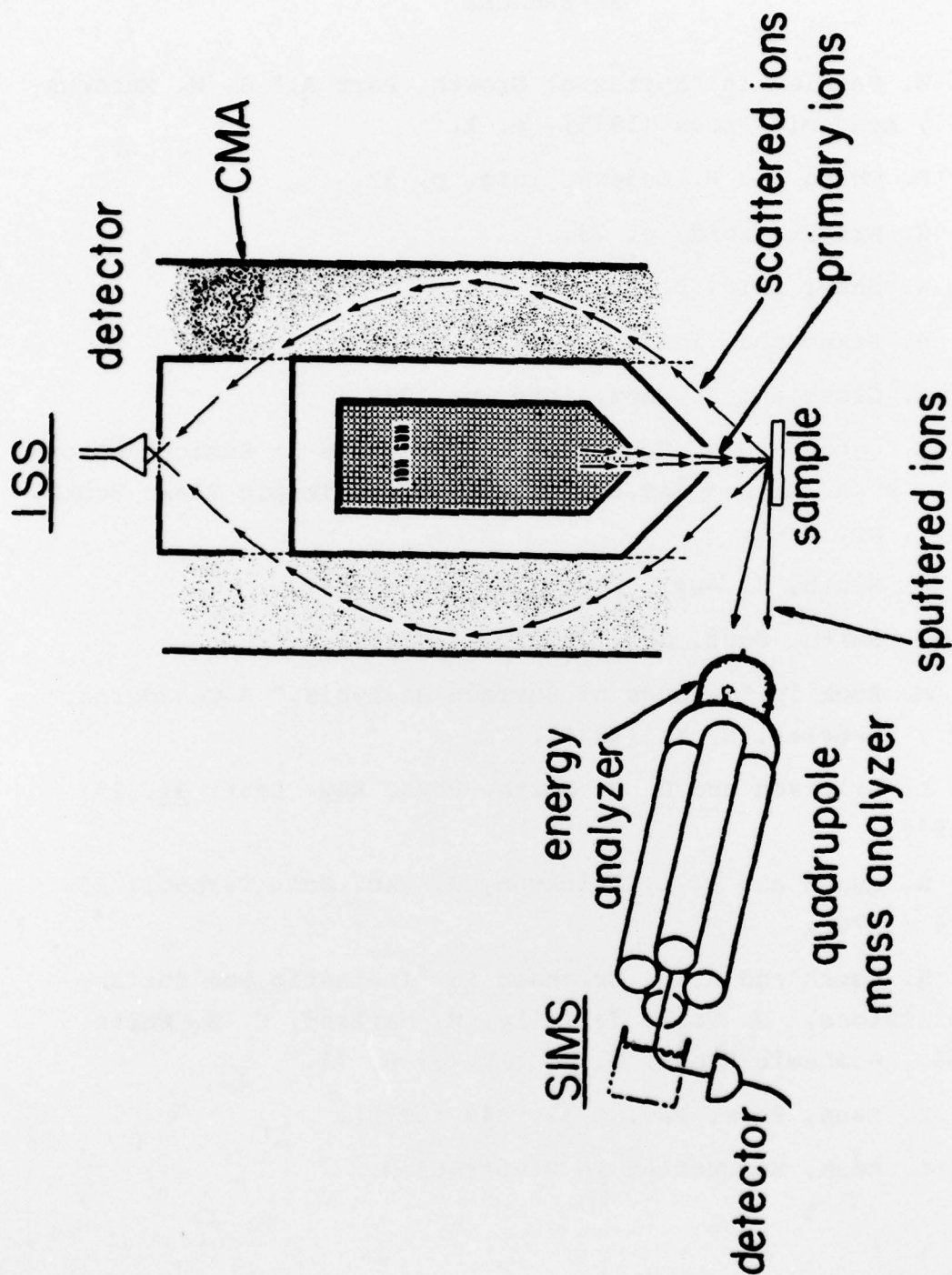


Figure 1. Components in UHV for Ion Scattering Spectrometry (ISS) and Secondary Ion Mass Spectrometry (SIMS)

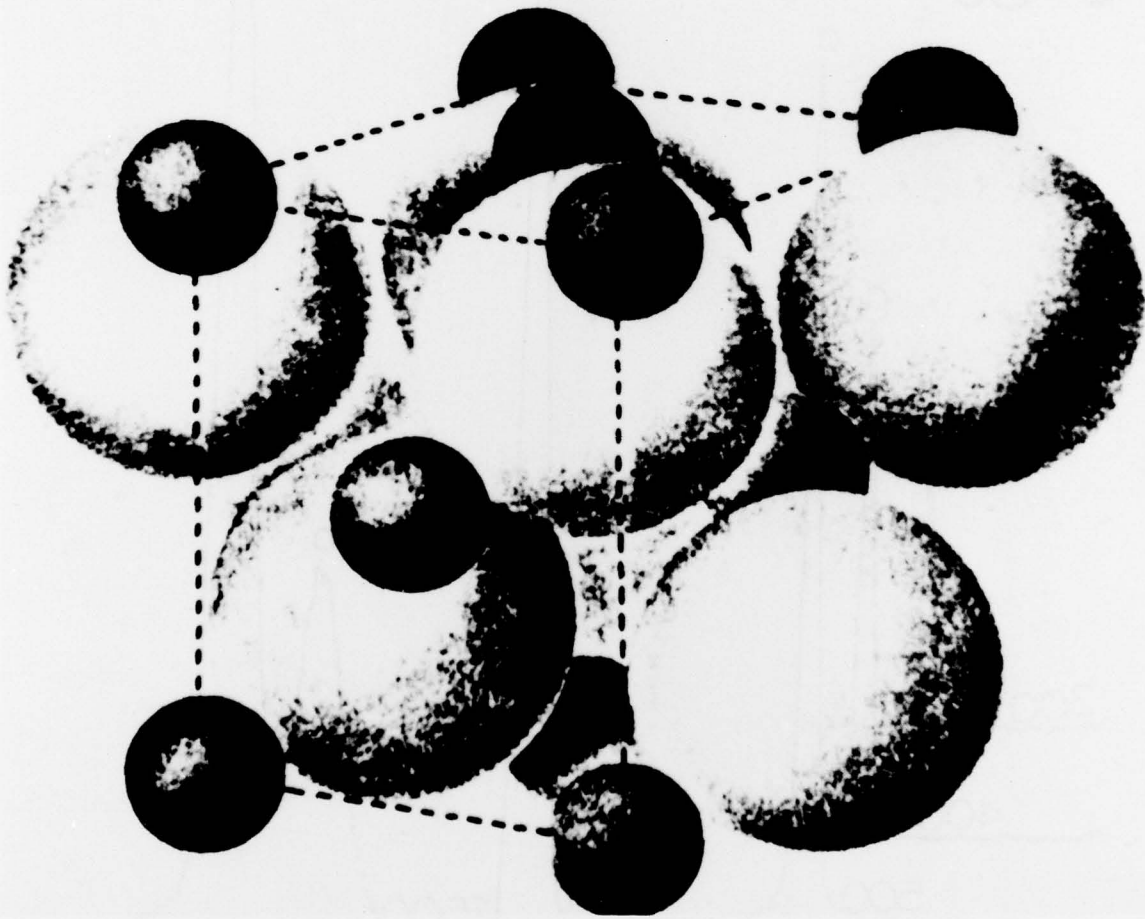


Figure 2. Sphalerite Structure Adopted by CdTe and HgCdTe

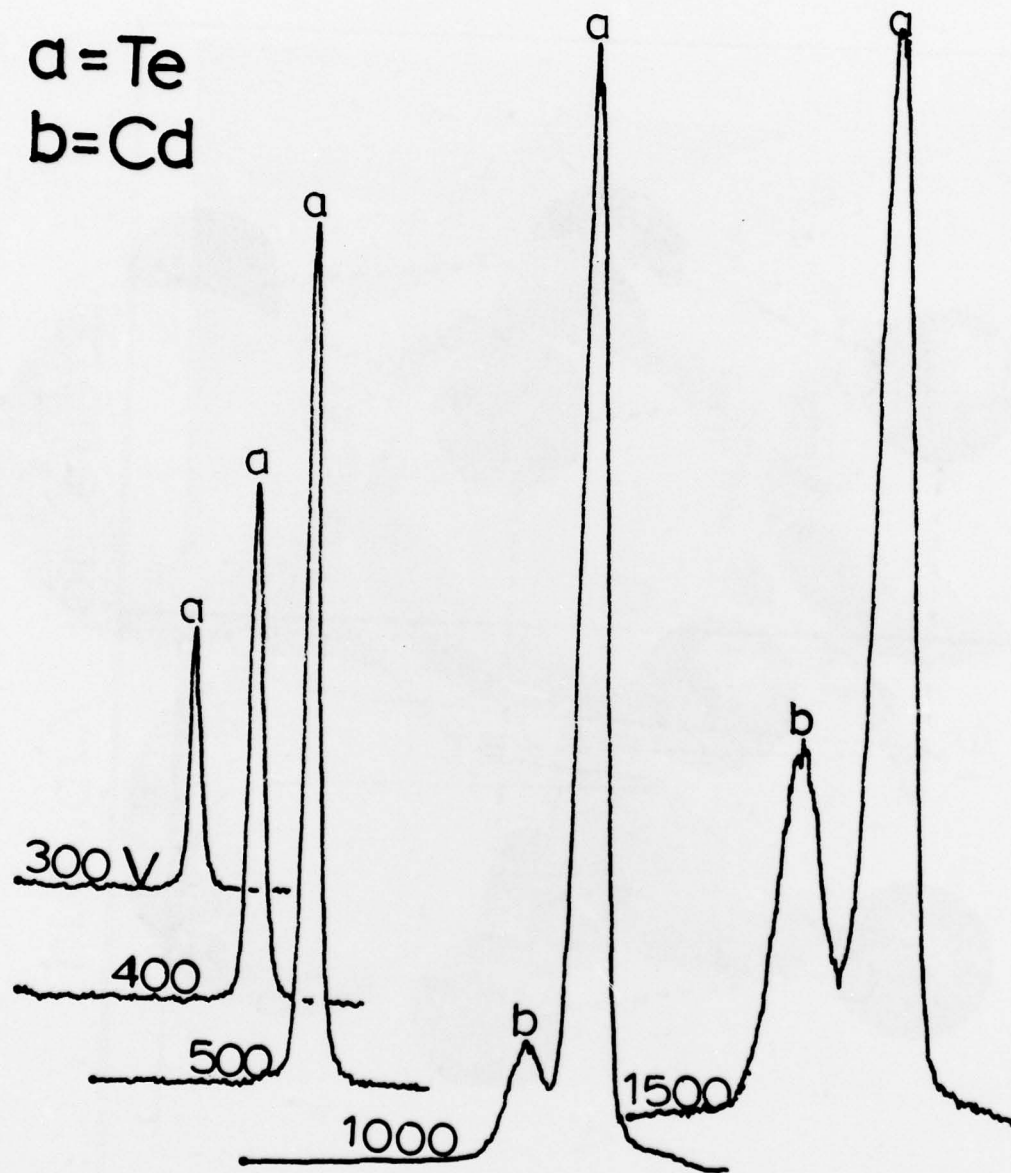
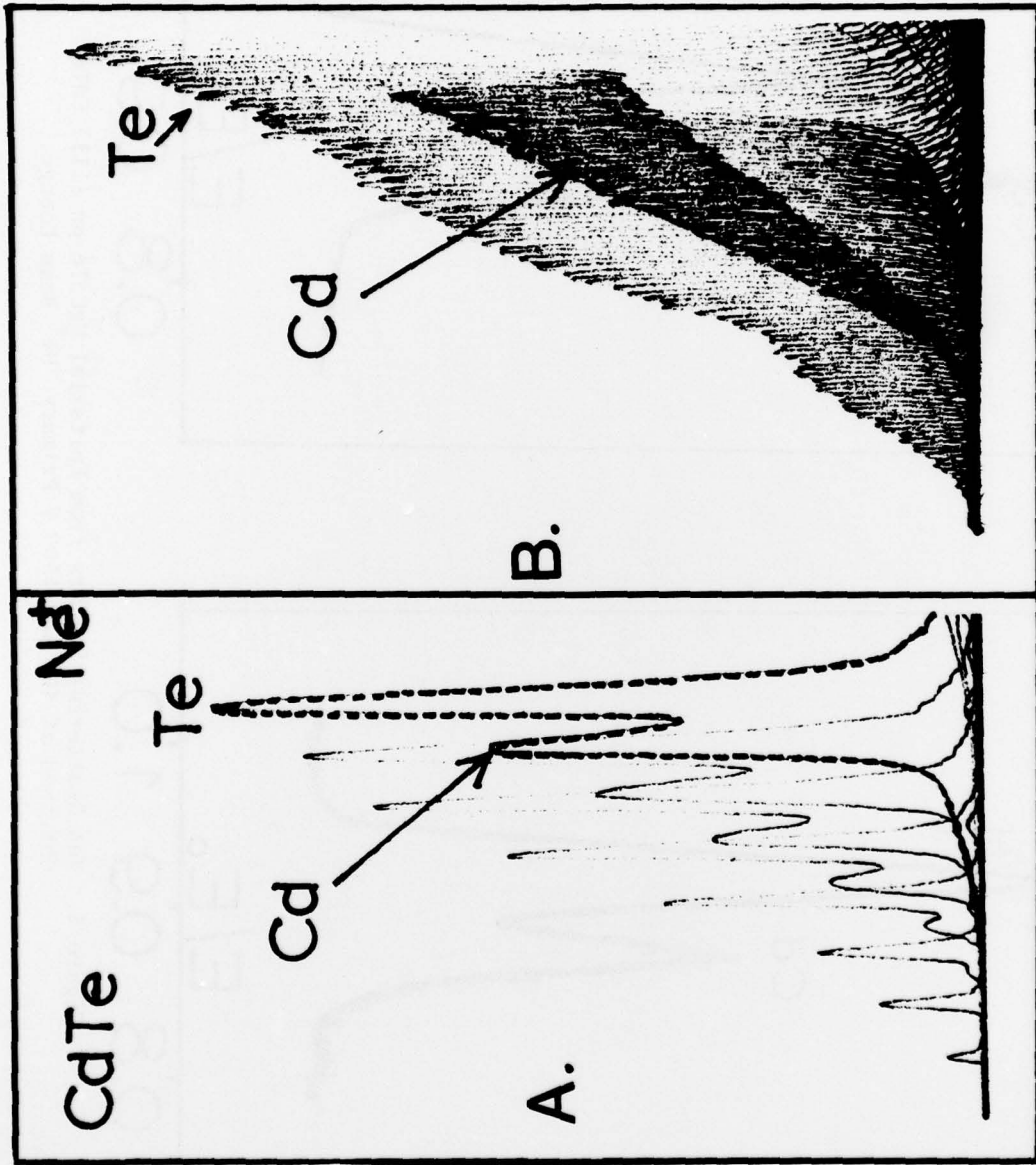


Figure 3. Neon Ion Scattering from CdTe (111)



primary ion energy

Figure 4. Ion Scattering Spectra 0-2500 V Using Ne^{20+} from {111} CdTe.
A. Repetitive Scans Obtained Slowly to Show Details of Spectral Changes with Increasing Energy. B. Repetitive Scans Obtained Rapidly.

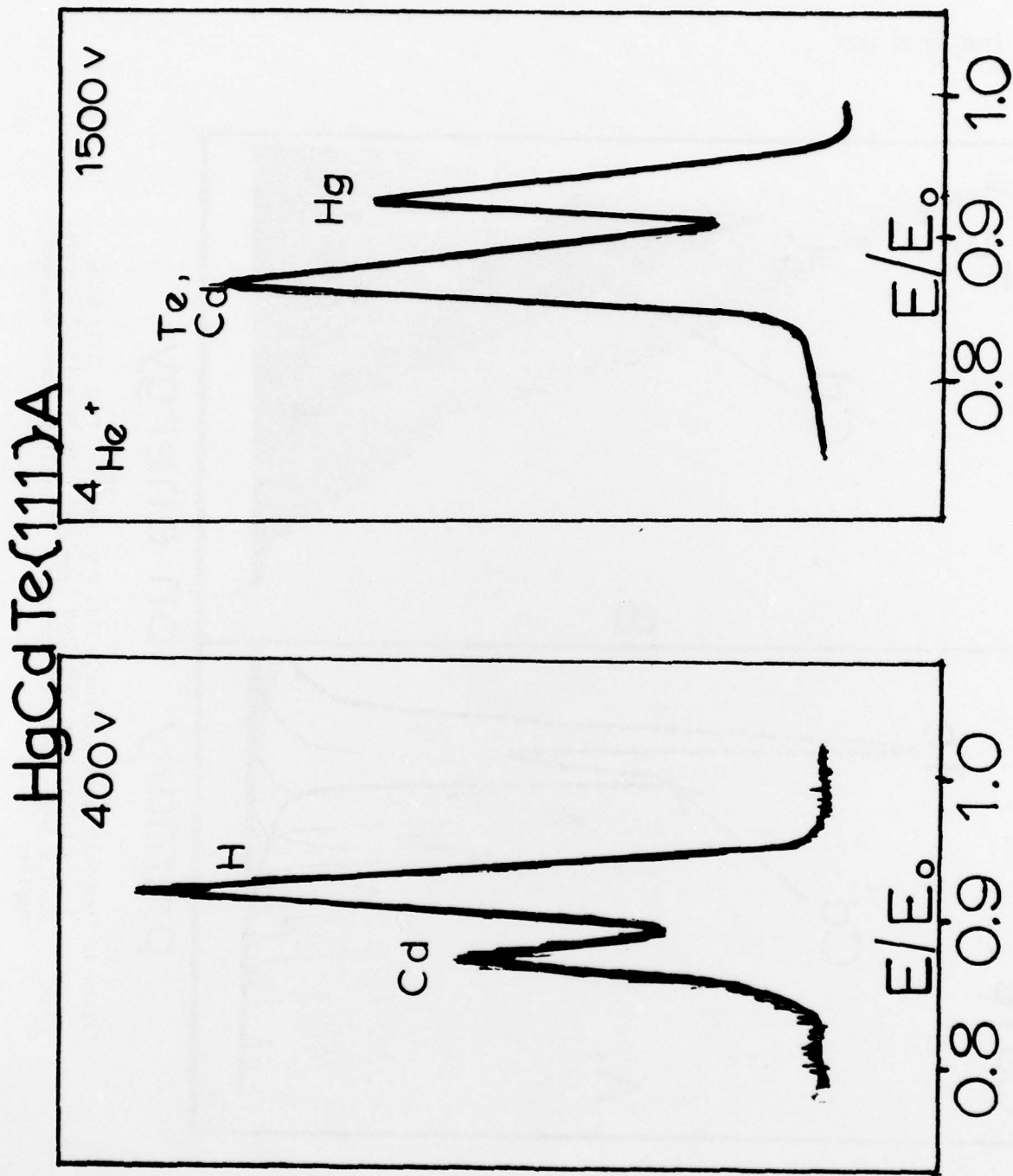


Figure 5. Ion Scattering Spectra from Epitaxial HgCdTe on (111) CdTe Obtained at 400 and 1500 V Primary $4He^+$ Beam Energy.

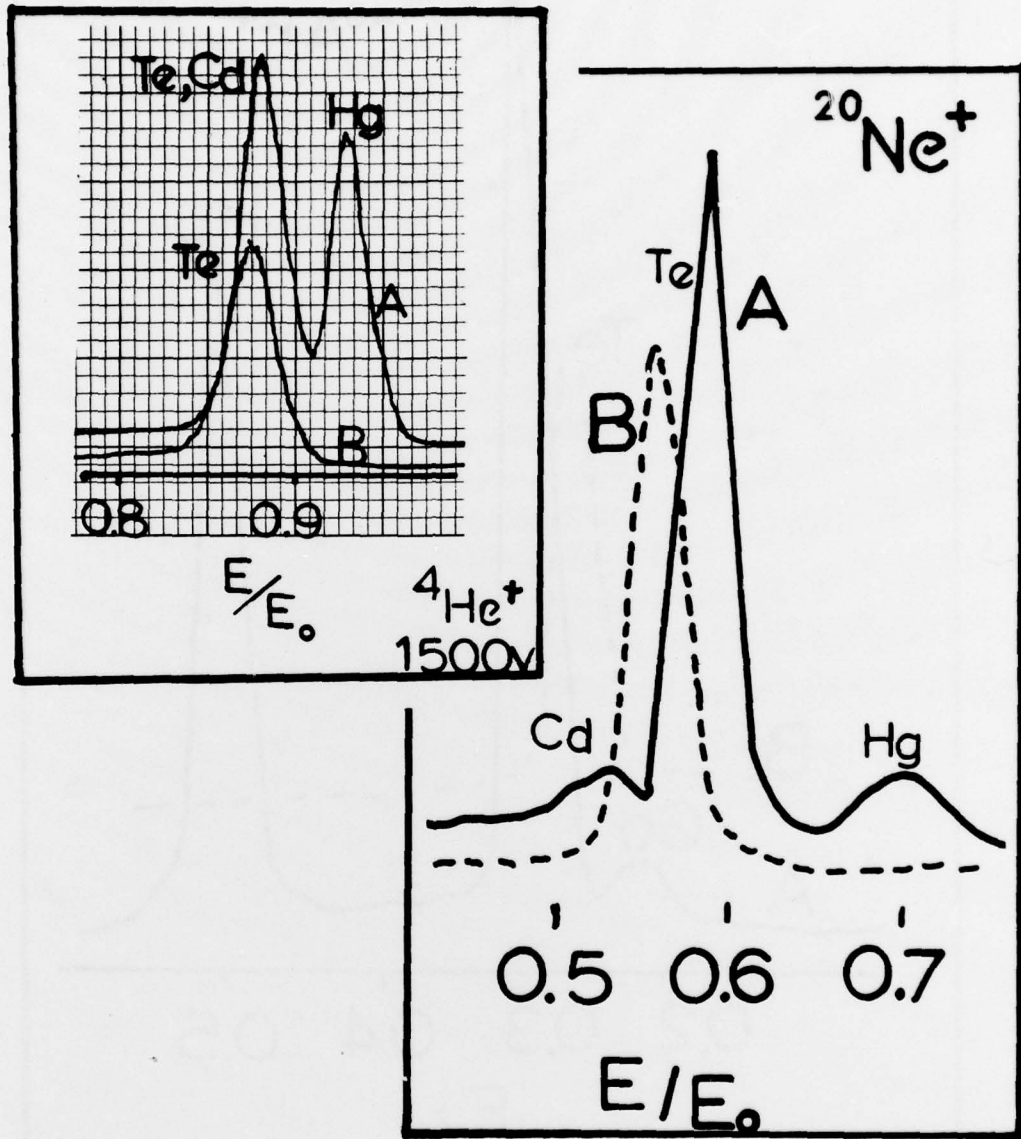
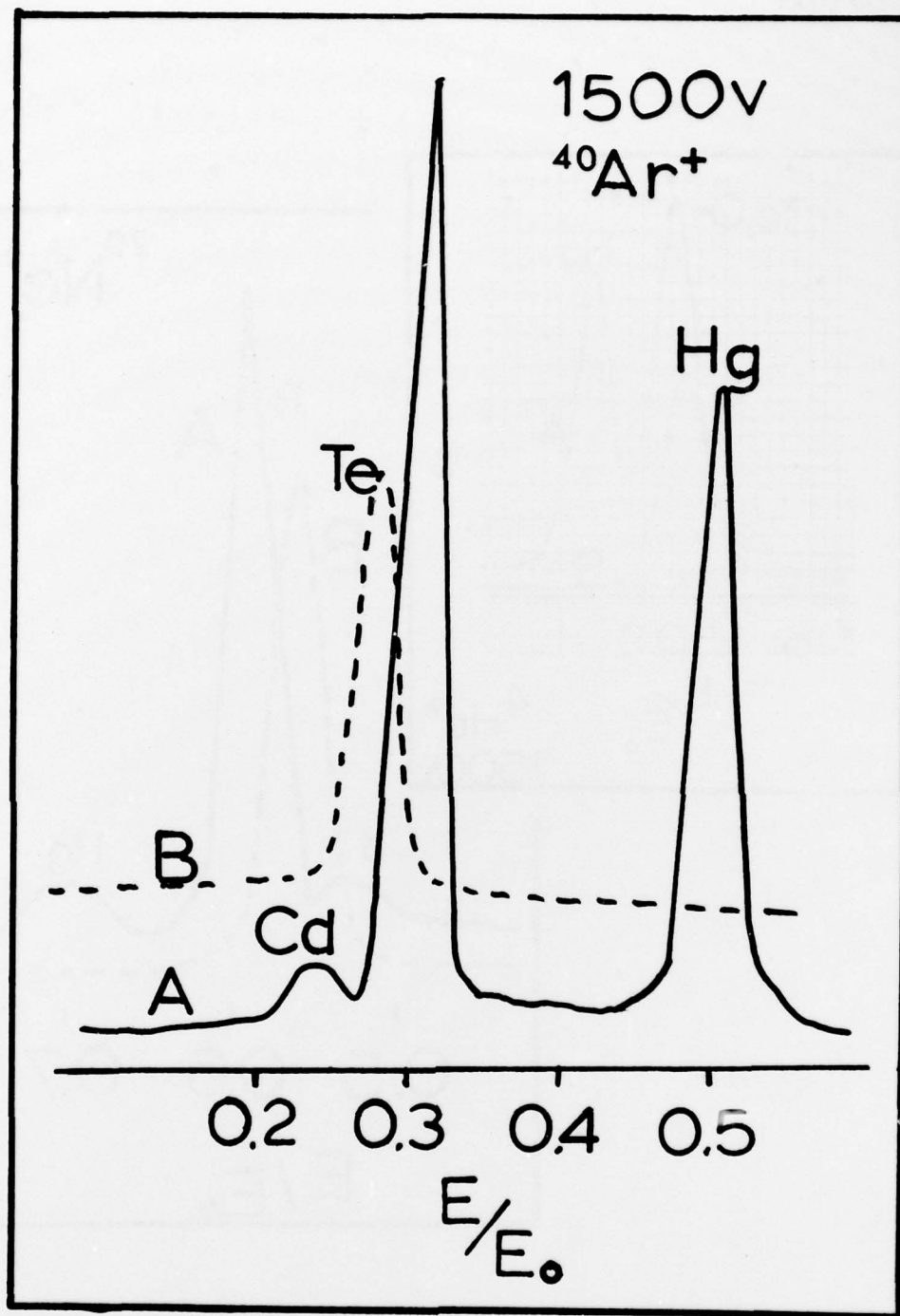


Figure 6. ${}^{20}\text{Ne}^+$ and ${}^4\text{He}^+$ Ion Scattering Spectra from {111}A and {111}B HgCdTe Epitaxially Grown on CdTe.



40 +
 Figure 7. Ar Ion Scattering Spectra from {111}A and {111}B Surfaces of HgCdTe Epitaxially Grown on CdTe.

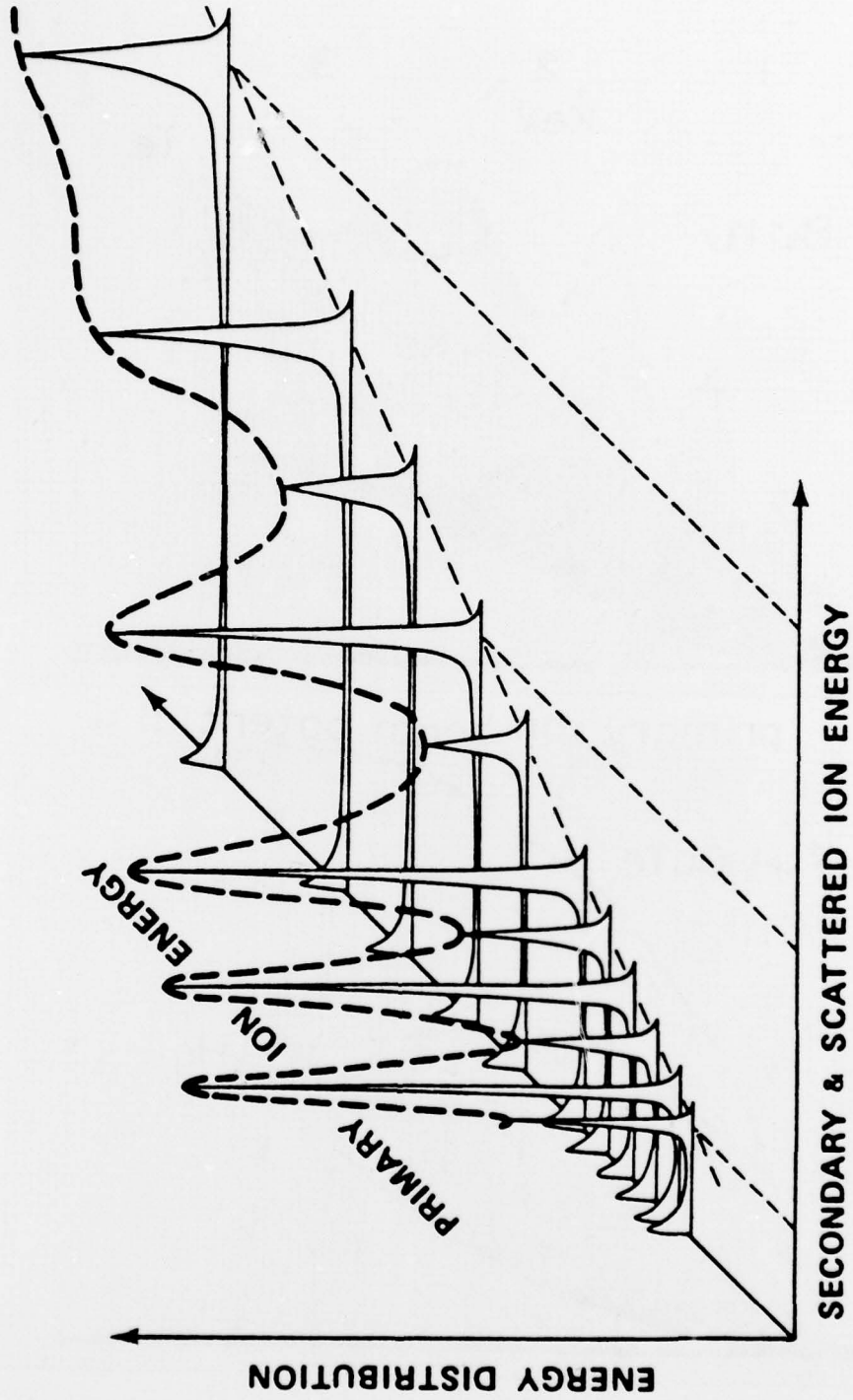
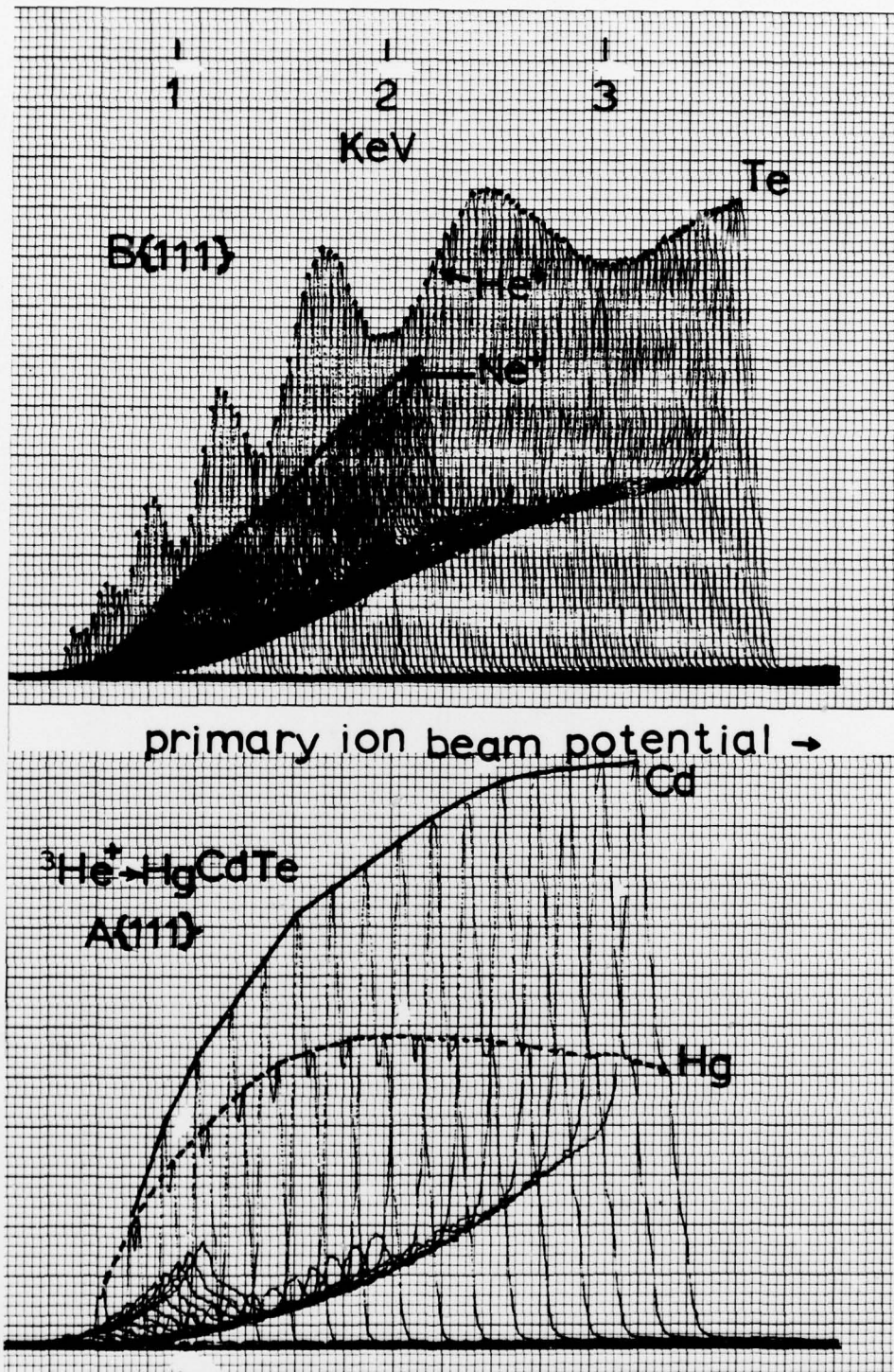


Figure 8. Repetitive Scans of He^4+ Ion Scattering Spectra from Scattered Pb Showing Method of Obtaining Yield Curve Structure (Reference 13)



3 +
 Figure 9. Repetitive Scans of He Scattering Spectra from {111}A and {111}B Faces of HgCdTe Showing Differences in Structure on Scattered Ion Yield Curves.

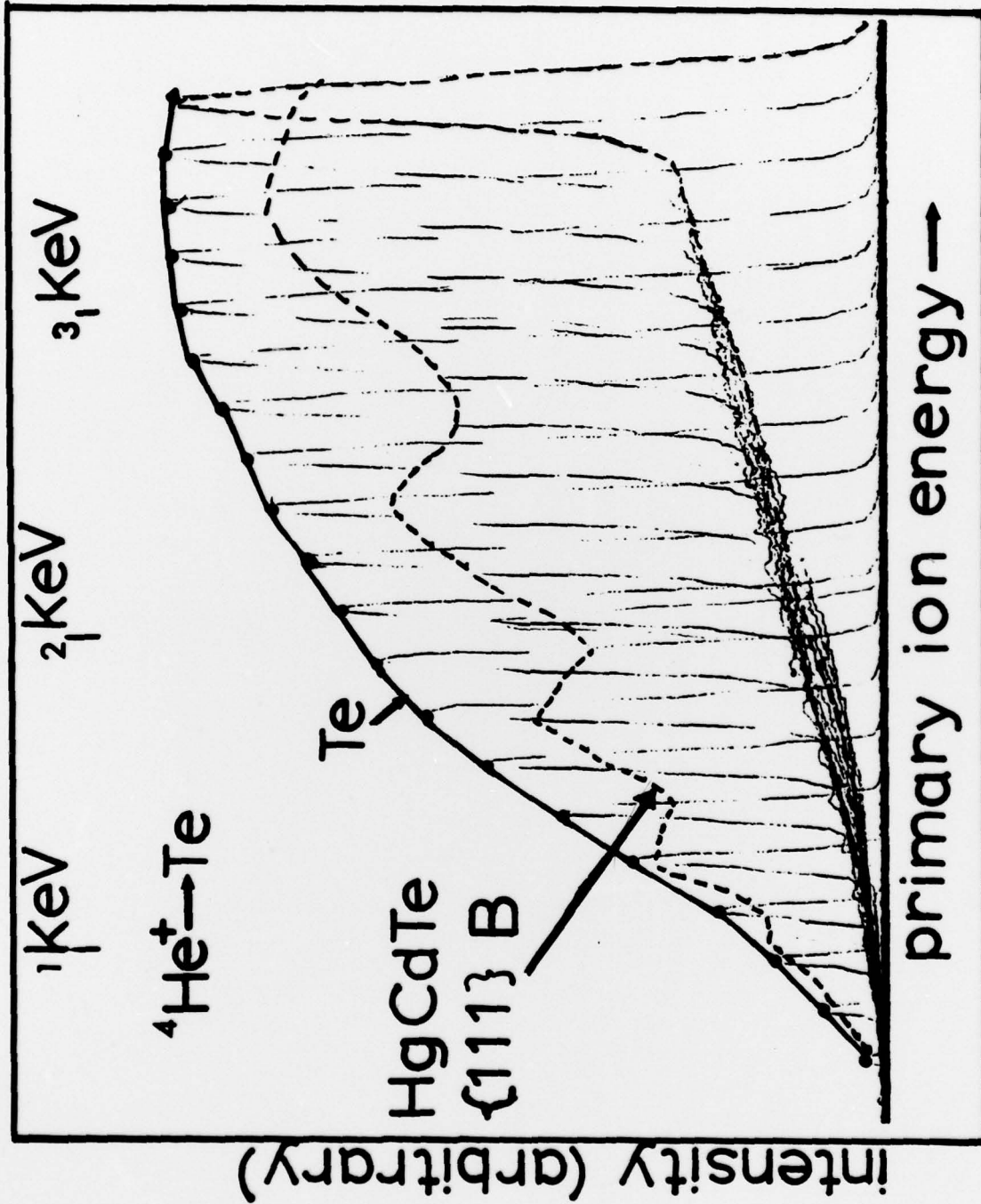


Figure 10. Repetitive Scans of the ${}^4\text{He}^+$ Ion Scattering Spectra from Elemental Te and from {111}B HgCdTe