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LIFE BEYOND 1MeV

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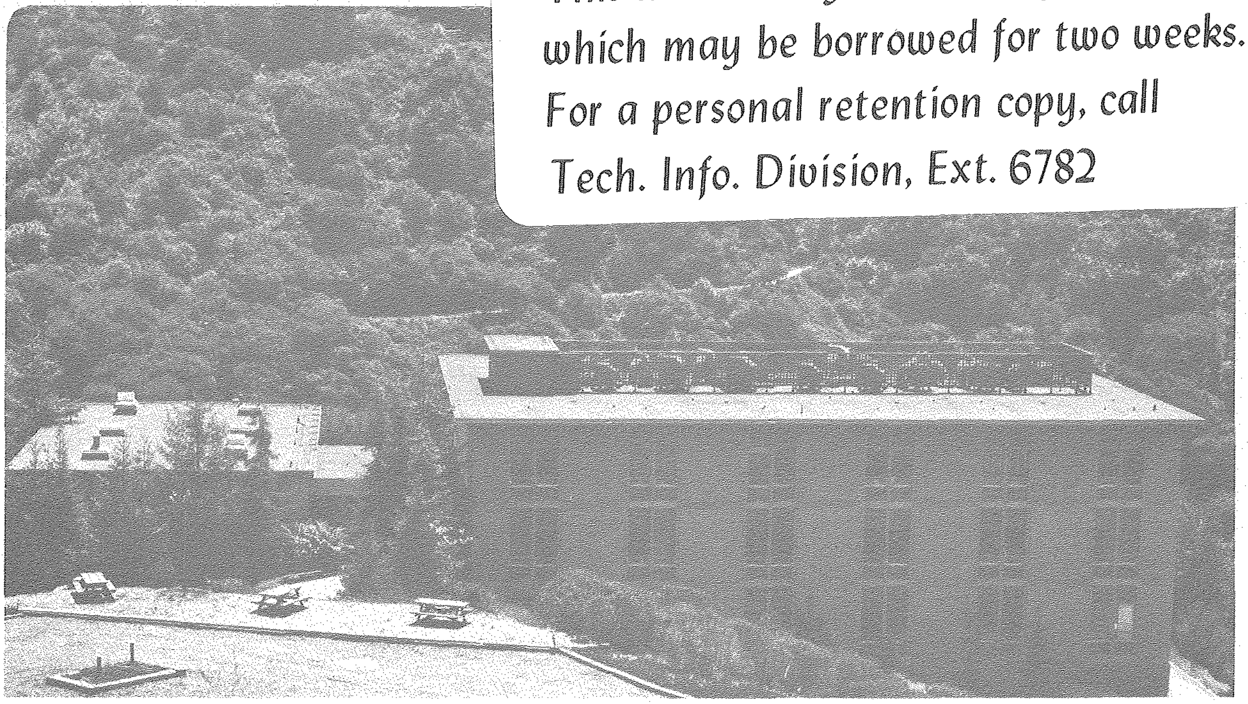

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DISCLAIMER

LIFE BEYOND 1MeV

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Life beyond 1MeV - like life after 40 - is not too different unless one takes advantage of past experience and is receptive to new opportunities. At first glance, the returns on performing electron microscopy at voltages greater than 1MeV diminish rather rapidly as the curves which describe the well-known advantages of HVEM often tend towards saturation. However, in a country with a significant HVEM capability, a good case can be made for investing in instruments with a range of maximum accelerating voltages. In this regard, the 1.5MeV KRATOS HVEM being installed in Berkeley will complement the other 650KeV, 1MeV, and 1.2MeV instruments currently operating in the U.S. One other consideration suggests that 1.5MeV is an optimum voltage machine - its additional advantages may be purchased for not much more than a 1MeV instrument. On the other hand, the 3MeV HVEM's, which seem to be operated at 2MeV maximum, are much more expensive.

Since the main rationale for constructing the original HVEM's was the increased penetration, this aspect will be considered first. In Fig. 1, results are collated from a number of studies of usable penetration as a function of accelerating voltage for several materials. For the purposes of comparison, the foil thickness at high voltage relative to that at 100kV is plotted. These results must be treated with some caution since the operating reflection and diffraction conditions were not always the same, and it is known that these are important parameters.[1,2] Nevertheless, the overall trends are reliable and it is clear that the advantages in going to 1MeV continue to 1.5MeV, but at a diminishing rate. Two points must be borne in mind, (1) experience has shown that bulk as opposed to thin foil behavior is observed in foils in excess of 1-3μm thick, and (2) dislocation contrast diminishes before the maximum foil thickness is reached. Thus, in studies of dislocation structures, foil thicknesses which are often less than the maximum will be naturally selected for observation. In other types of experiments, however, the use of very thick foils is a decided advantage. For example, during in-situ oxidation studies of thin foils, selective oxidation of one component of an alloy can lead to severe solute depletion of the matrix and non-bulk behavior. Secondly, the very high penetration in the light elements, such as Si, facilitates direct observations of interfaces, defects, etc., in materials for electronic devices and solar energy applications where layer thicknesses are of the same order as foil thicknesses.

A second important advantage of HVEM's is the greatly increased space in the specimen chamber. This allows construction of "mini-labs" in the objective pole piece for performing a variety of in-situ studies. This aspect has already been extensively developed (see e.g., Butler[3]) and, with the great current interest in understanding the behavior of materials at high temperature and in hostile environments, is likely to be pursued further in the next decade. In particular, by sacrificing some of the increase in penetration at high voltages and surrounding the specimen with a gaseous environment of controlled composition, a much better understanding of gas-solid reactions and interface behavior can be anticipated. Swann was one of the early developers of environmental cells for such studies, and in Fig. 2 Swann and Tighes'[4] experimental data of gas pressure as a function of accelerating voltage for several gases have been extrapolated to 1.5MeV. The criterion that the maximum usable pressure, P_V, is that at which $I/I_o = 0.1$ was used, and the pressure relative to P_V at 100kV is again plotted. For some gases the increase in pressure in going to 1.5MeV is appreciable, and should be feasible. Under normal (non-environmental cell) use the column vacuum is also important, and consequently, the Berkeley instrument has been equipped with more efficient 500ℓ/sec turbo-molecular pumps. The attainable vacuum is $\sim 10^{-6}$ torr in the specimen chamber (this can be improved further by auxiliary ion-pumping on a port in the chamber), and 10^{-7} torr at the base of the accelerator tube. A good accelerator vacuum is also desirable, since it is known that the time interval between accelerator conditioning is increased and the X-ray emissions decreased with improvement in vacuum.

The ability to simulate radiation damage in an HVEM has been exploited extensively in the past and is likely to continue. However, many of the heavier elements, e.g., W, Au, U have threshold energies for displacement damage, E_T, in excess of 1MeV and could not therefore be studied in this country. At 1.5MeV, E_T is exceeded for all the elements in the periodic table, and this added capability could be an asset in future work. With increased emphasis on fusion systems which operate at higher temperatures and require new classes of materials, refractory metal alloys which have high E_T's (e.g. Mo = 870KeV), will find increased application. Unfortunately, radiation damage is a double-edged sword and in many applications of high voltage microscopy it is an unwanted complicating factor in the experiment. Two solutions to this problem have been suggested. Operation at a voltage below E_T - not good unless E_T is high, or perform the experiment at a temperature where the thermal equilibrium concentration of vacancies exceeds the concentration of displaced atoms produced under the conditions of the observation. Values of these temperatures for a number of metals have been given by Bricknell and Edington.[5] Fortunately, this second option is more compatible with in-situ high temperature studies of materials at high accelerating voltages.

Perhaps the greatest future potential for HVEM's, however, lies in the inherent capability for improvements in resolution, i.e., the $C_s\lambda$ effect. Horiuchi et al.[6] have graphically demonstrated this potential with excellent "structure images" taken at 1MeV with axial illumination on evaporated gold film. Lattice fringe images of the 2Å spaced (200) planes are clearly resolved. High resolution was a high priority specification for the Berkeley HVEM and the instrument was designed to achieve (with modification) 3.5Å point to point ($\sim$2.5Å lattice) resolution. In order to achieve these resolution levels, several design improvements have been incorporated in the instrument. The high voltage generator was built by the Emile Haefely Co. to more stringent specifications than earlier models. Wire wound resistors are used in the voltage divider and together with other changes improve the voltage stability to $<5 \times 10^{-6}$/3 min. at all the fixed voltages from 200-1500kV. The ripple voltage between 500kV and 1500kV is $<3 \times 10^{-6}$. A detailed description of the system has been given by Reinhold.[7] The objective lens is designed with a high-resolution pole piece and top entry stage as well as the usual side entry system, and the lens current stability is better than 3×10^{-6}/3 min. A magnification range from 50X to >630,000X is provided with rotation-free imaging above 2500X.

In addition to instrumental changes, the building to house the microscope has been designed to prevent ambient ground vibrations, and those resulting from future site development, from limiting the instrument performance. The microscope is mounted on a 100 ton inertia block (of 1.2Hz natural frequency) which in turn is supported on 12 air springs. Because of the vulnerability of its location, an additional 4 seismic restraint devices are fitted which activate to restrict block motion in the event of an earthquake. The system is designed to protect the installation from major damage in a 7.0 earthquake with epicenter 0.5 mile from the building (e.g., on the Hayward Fault). A schematic diagram of the vibration isolation system is shown in Fig. 3, and a panoramic view of the microscope tower and support facilities is given in Fig. 4.

A diverse research program is planned for the new instrument including in-situ studies of gas-solid reactions and phase transformations, studies of ceramics, glasses, minerals and other beam-sensitive materials, microstructure characterization of steels, solar cells, etc. In these studies conventional HVEM imaging techniques will be augmented with information from energy loss spectroscopy and convergent beam microscopy.

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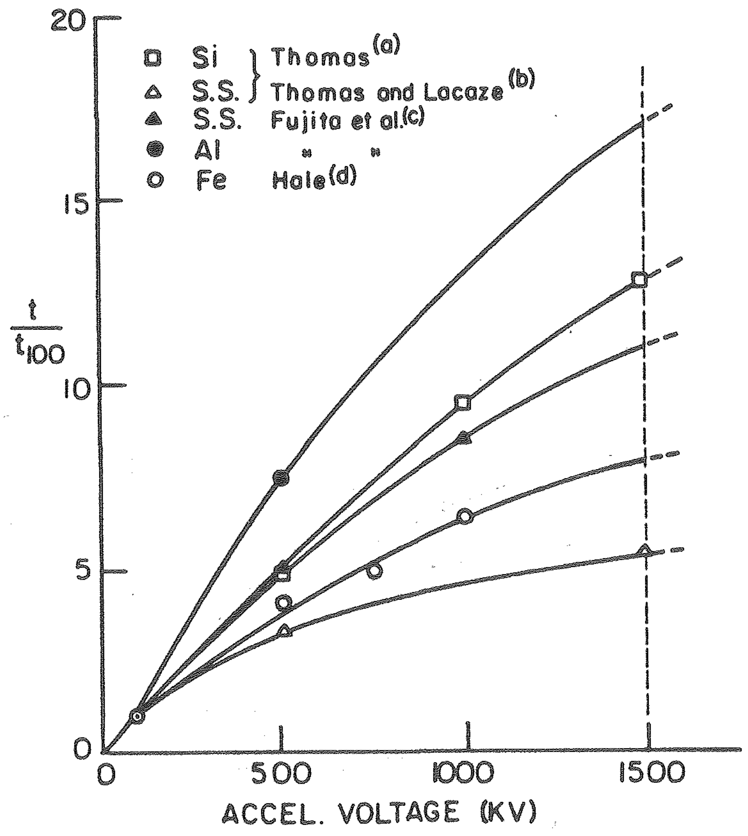

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Figure 1

Plot of maximum usable foil thickness (relative to 100KV) versus accelerating voltage for Si, Fe, stainless steel.

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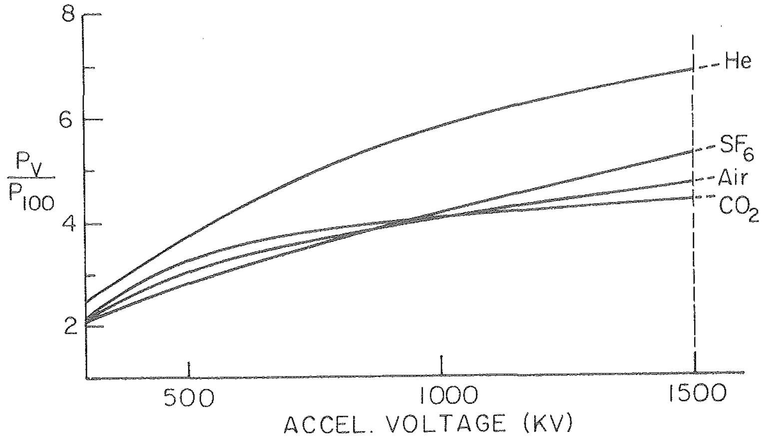

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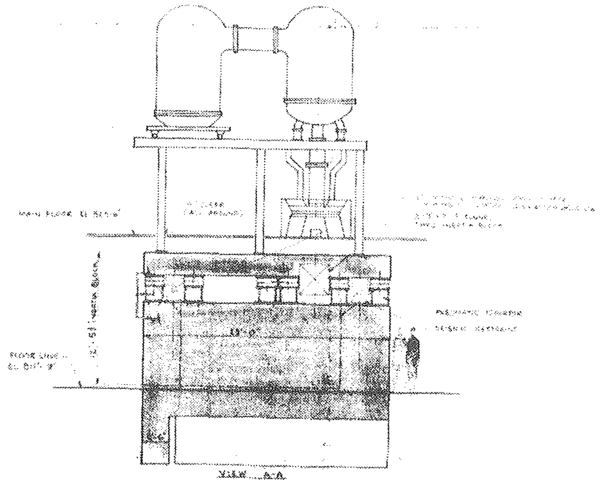
Figure 2
Plot of maximum usable gas pressure (relative to 100KV) versus accelerating voltage for several gases. (Data of Swann and Tigue[4]).

Figure 3
Schematic diagram showing vibration isolation foundation for Berkeley 1.5 MeV HVEM.



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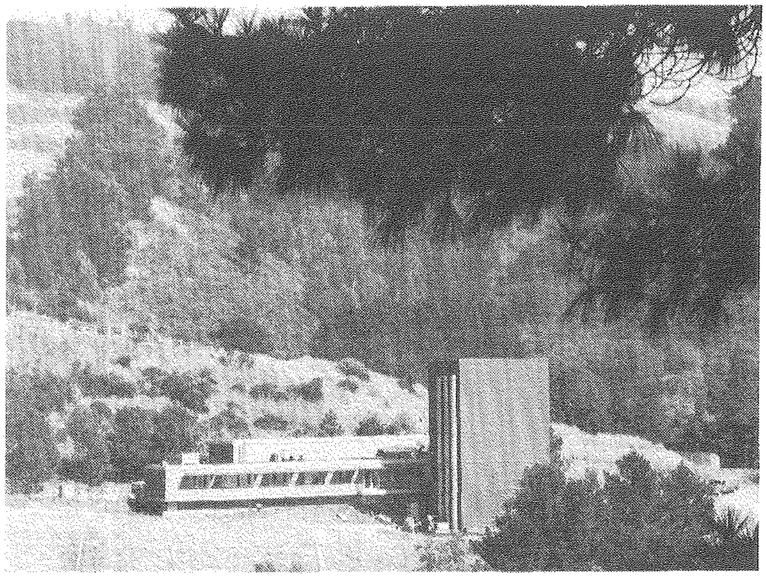

Figure 4
Photograph of Lawrence Berkeley Laboratory 1.5 MeV High Voltage Electron Microscope facility.

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