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High Voltage and High Resolution Electron
Microscopy in Materials Science

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1. Introduction

There is no doubt that transmission electron microscopy has made a dramatic impact on metallurgy and materials science over the past twenty years and the literature certainly reflects this growth. In this time we have seen a prolific burst of microstructure information which is only now being utilized directly for engineering applications e.g. alloy design. It is easy to forget that it was only twenty years or so ago that we first saw dislocations moving in an electron microscope and that the first significant publication on transmission electron microscopy was that of Heidenreich in 1949⁽¹⁾.

The field of electron microscopy continues to grow with exciting developments occurring in several areas, e.g. high voltage, high resolution and analytical microscopy. Resolutions of atomic dimensions have recently been reported in a remarkable result of Hashimoto⁽²⁾ in which the atomic arrangement in a [001] gold crystal has been imaged directly (Figure 1). This is not a routine experiment as yet and we are a long way from equivalent resolutions in spectroscopy for elemental analysis, although energy loss spectroscopy at high voltages in transmission through thin samples shows great promise for such achievements⁽³⁾. Other developments using fine focussed beams for micro-diffraction and energy dispersive analysis such as in STEM instrumentation have indicated considerable advances for analytical applications, as discussed in the article by Maher.

In the present paper a brief review will be given of the principles and some applications of transmission electron microscopy at high voltages and high resolutions. The combination of high resolution electron imaging and laser optical microdiffraction shows great promise for lattice parameter measurements over localized regions ($\sim 10\text{\AA}$) from which estimates of composition changes can be made.

2. Basic Background

The information that is obtained by electron microscopy methods is derived from the scattering processes that take place when the electron beam travels through the specimen. There are two main types of scattering (a) elastic - the interaction of the electrons with the effective potential field of the nuclei - involving no energy losses and which can be coherent or incoherent (poor phase relationships) (b) inelastic - the interaction of the electrons and the electrons in the specimen involving energy losses, i.e., absorption. It is the elastic scattering that produces a diffraction pattern; and if the scattering centers in the specimen are arrayed in an orderly, regular manner such as in crystals, the scattering is coherent and results in spot patterns, Kikuchi patterns and, if the sample is a fine-grained polycrystal, ring patterns. Thus the information from electron microscopy includes morphology (images) structure (diffraction), and chemical analysis (spectroscopy) as indicated in Figure 2. The electron microscope is truly a versatile instrument.

The basic reason for the utilization of the electron microscope is its superior resolution resulting from the very small wavelengths of electrons compared to other forms of radiation for which an optical system can be constructed. The resolution is given by the Rayleigh formula which is derived from considering the maximum angle of electron scattering (α) which can pass through the objective lens. This formula is:

$$R = \frac{0.61\lambda}{\alpha} \quad (1)$$

where R is the size of the resolved object, λ is wavelength, and α is identical to the effective aperture of the objective lens.

In the electron microscope, the effective aperture is limited chiefly by spherical aberration. The spherical aberration error is:

$$\Delta S = C_s \alpha^3 \quad (2)$$

where C_s is the coefficient of spherical aberration of the objective lens ($\approx$ focal length which is about 2 mm or less in modern instruments).

Thus R increases with decreasing α ; whereas ΔS decreases with decreasing α .

As a result, in electron optics one arrives at an optimum aperture and minimum resolution given by:

$$\alpha_{\text{opt}} = A(\lambda^{1/4})C_s^{-1/4}, \quad (3)$$

$$\Delta R_{\text{min}} = B(\lambda^{3/4})C_s^{1/4} \quad (4)$$

where A, B are constants of order 1.

The relativistic wavelength of electrons depends on the accelerating voltage and is given by the modified DeBroglie wavelength; which reduces to:

$$\lambda = \frac{12.26}{E^{1/2}(1+0.9788 \times 10^{-6}E)^{1/2}} \text{ \AA} \quad (5)$$

where E is the accelerating potential (volts), and thus λ decreases with E.

Some values pertinent to electron microscopy are given below.

E(volts)	$\lambda(\text{\AA})$	$\lambda^{-1}(\text{\AA})^{-1}$	$(v/c)^2$
100kV	0.037	27.02	0.3005
500kV	0.0142	70.36	0.7445
1MeV	0.0087	114.7	0.8856
10MeV	0.0012	846.8	0.9976

Another advantage of the small wavelength of electrons is that the depth of field and depth of focus are very large in electron microscopes.

At 100kV the point resolution $\Delta R_{\min} \approx 3.5\text{\AA}$ for $C_s = 1.6 \text{ mm}$. Other factors which affect resolution are astigmatism and chromatic aberration of the imaging system, and chromatic aberration resulting from energy losses in the specimen. These errors produce poor resolution for non-axial illumination. However, since C_s decreases with voltage in theory we should be able to attain point resolutions better than $2\text{\AA}$ provided adequate technical and mechanical stability is achieved, by raising the voltage to 0.5 or 1MeV. Attempts in this direction are well underway in various parts of the world, but not unfortunately in the USA. However, experimental HV STEM systems are being developed at Chicago and Arizona.

The conventional imaging mode for TEM is either bright field (transmitted electrons) or dark field (diffracted electrons). However, if the aperture of the objective lens is large enough to allow recombination of the transmitted and diffracted beams a lattice image is obtained which is a magnified picture of the planes which are diffracting. At the present time the resolution for this mode of imaging is about $1.5\text{\AA}$. Applications of this method are discussed later.

Unless one utilizes a very fine probe, as in STEM, then, because of spherical aberration, the error in selection of the area for diffraction analysis can be appreciable. The minimum selected area ΔA is given by Eq. (2), replacing α by the Bragg angle θ , i.e.,

$$\Delta A \approx C_s \theta^3 \approx C_s (\lambda/d)^3 \quad \text{for small angles.}$$

Since C_s varies approximately as λ^{-1} then ΔA varies as λ^2 and thus decreases rapidly with increasing voltage. For example for a fixed beam TEM, $\Delta A \approx 2\mu\text{m}$ at 100kV and $0.02\mu\text{m}$ at 1 MeV. This illustrates another advantage of high voltage microscopy. The smallest size aperture D normally used is $5\mu\text{m}$ and considerable care must be exercised so as to align the microscope correctly with proper focusing.

The combination of HVEM with STEM where a fine focus intense coherent beam can be achieved should provide even better combinations of resolution and analytical features. Thus, for microdiffraction and analysis of small particles this approach should be particularly advantageous, so we watch with interest the developments at Chicago and Arizona.

3. High Voltage Microscopy

From the foregoing the advantages of high voltage electron microscopy over conventional (100kV) microscopy are apparent. We define HVEM as microscopy at 500kV and above. Table 1 lists these advantages whilst Table 2 lists some of the disadvantages - the chief one being the high cost (approximately \$1 per volt, but rising!). At the present time there are 10 HVEM's in the USA with two HVEM-STEM microscopes under construction at Chicago and Tempe. The only ultra-high HVEM's are abroad in Toulouse and Osaka (both with 3MeV capabilities).

The main applications in materials science utilize the improvements in penetration, and resolution, and particularly for using the microscope as a mini-laboratory for in-situ studies (Figure 2) although again the US is somewhat behind the rest of the world in this regard. Due to the gain in penetration with voltage, materials difficult to thin are now achieving considerable attention

e.g. ceramics and minerals. Experimentally it is found that the most significant gain in penetration is for light elements, Si, Al (see Table 1).

The reduction in inelastic scattering with increasing voltage implies a reduction in ionization and other damaging processes, and this has been observed for several biological and polymeric solids. However, knock-on damage occurs above a threshold energy which is roughly proportional to atomic number (e.g. $\approx 500\text{keV}$ for copper; 1MeV for gold).

As a consequence, a considerable effort is being made to simulate neutron radiation damage by HVEM and because of the high flux in the latter, point defect production rates are orders of magnitude faster than in a reactor. Thus the problem of swelling may be eventually understood by continued efforts involving HVEM and other simulation techniques (ref. 4).

On the other hand, the reduction of ionization damage allows the electron microscopy of beam sensitive materials (polymers, biological specimens, certain ceramics, and minerals, etc.) to be done. Recent work done on the aliphatic amino acid l-valine⁽⁵⁾ has shown that the critical exposure (viz. the electron flux necessary to destroy crystallinity of a material) varies with energy as β^3 ($\beta = v/c$ where v is electron velocity, c is velocity of light) rather than as β^2 as predicted from stopping power theory. A consequence of this gain in specimen stability is a gain in resolution of about a factor of x2 in operating at 1MeV rather than 100kV . That is, the severe limitation on resolution imposed by radiation damage is greatly alleviated at high voltages.

As can be seen from Table 1, the advantage and range of applications of HVEM are many indeed and there is no space here to cover them all in detail (see e.g. references 6-9). Brief mention of some current applications at Berkeley follow.

Table 1. Some main advantages of high-voltage transmission electron microscopy

1. Penetration

Light materials $\approx 14 \mu\text{m}$ at 2.5 MeV

Medium-heavy $\approx 2-3 \mu\text{m}$ at 1 MeV (maximum kV for routine microscopy)

Applications include:

Biological systems-natural environments, thick sections

Ceramics, minerals, difficult to thin materials

Nondestructive, precious samples, e.g., moon dust

Particle analysis (extractive metallurgy), pollution research

Heavy elements

Semiconductor devices

Crystals of low defect densities

Better statistics-larger sampling (stereomicroscopy)

In-situ studies (dynamic events), environmental; gases, liquids, etc.

Interfaces, e.g., diffusion gradients, semiconductors, multiphase systems

2. Resolution Increase

Chromatic and spherical aberrations

Reduced area for selected area diffraction

Lattice defects

3. Special Diffraction Effects—Crystals

Channelling increased transmission

Critical voltage effect

Contrast: Improved magnetic contrast

Improved resolution of lattice defects (bright field imaging using high order systematics)

4. Displacement Damage

Simulates nuclear reactors, e.g., at 1 MeV point defect damage in Fe in electron microscope in 1 min $\approx$ 450 days in reactor

Maximum voltage should be about 1.5 MeV (1.25 MeV is threshold for uranium)

5. Ionization Damage

Decreases with increasing kV (e.g., beam-sensitive materials—biological, polymers, ionic crystals, etc.)

6. Superior Vacuum

Lower contamination rate

7. Higher Brightness

Table 2. Some disadvantages of high-voltage transmission electron microscopy

1. High cost and maintenance (capital costs are $\approx 10^6$ dollars for 1.0 MeV microscopes).
 2. X-ray hazard (can be avoided by careful screening, but needs monitoring).
 3. Radiation Damage to Specimens
Knock-on damage in crystals destroys substructure, limits maximum kV usable, therefore, limits penetration advantages.
 4. Manufacturing Defects
Need for better, high quality commercial specimen stages, e.g., goniometer stage for range of temperatures and environments. Control of atmosphere is essential for in-situ work.
-

Phase Transitions and Magnetic Properties of Ferrites

The HVEM is especially useful for in-situ, dynamic studies (Fig. 2). Control of the environment is especially important in heating experiments, otherwise problems such as oxidation and contamination (e.g. with steels), and even reduction of oxides can occur in "normal" microscope atmospheres. Amongst studies of interest at Berkeley is the order $\leftrightarrow$ disorder transition in lithium ferrite. In situ experiments were done at Imperial College, London, but we plan to modify the Berkeley microscope to do such studies in the future. The ordering reaction occurs near 750 °C and the results are consistent with the theory of homogeneous nucleation and perfectly spherical growth. On the other hand, the disordering appears to start at the APB's.* Plans for incorporating environmental chambers at other HVEM installations, e.g. Case, U.S. Steel, and Richland are now well underway so more work in this area will appear in the future.

In addition to this order-disorder transition, at higher temperature there occurs a precipitation reaction. This transformation has been studied at 1200 °C in air, oxygen and vacuum. Coherent octahedral precipitates of LiFeO_2 form uniformly for short annealing times (Fig. 3). These precipitates become semi-coherent and grow anisotropically. Finally, material loss takes over and the LiFeO_2 particles transform back to spinel leaving behind small angle boundaries. The accompanying changes in the magnetic hysteresis curves (Figure 3) show that for the coherent two phase LiFeO_2 - LiFe_5O_8 mixture, the magnetic properties are improved⁽⁸⁾. These preliminary results suggest new research directions which may lead to advanced magnetic materials for electromagnetic communication and storage devices.

Defects: Except for simple structures, lattice defects in oxide ceramics have been somewhat neglected by ceramists. Dislocations play a fundamental role in the mechanical behavior of the oxide materials, as they do in metals, and the details of the dislocation structures should be of great importance with regard to high temperature deformation, creep, fracture, etc. Stacking faults also

* A 16 mm movie (sound) film of these processes is available on request.

affect magnetic properties. Detailed contrast experiments have therefore been carried out to characterize dislocations and stacking faults in CoFe_2O_4 and LiFe_5O_8 , using the special advantages of high order bright field systematic diffracting conditions to reduce image width and improve resolution⁽⁹⁾. This method is equivalent to the weak beam D.F. method used at 100kV. Figure 4 shows the dissociation of a dislocation to two partials imaged in high order bright field dynamical conditions. The need for detailed image profile computation for the complex materials is apparent from the figure. From such analyses the surface energy associated with the [110] cation faults has been found to be $\sim 75 \text{ ergs/cm}^2$. Other advantages of using high order bright field imaging and special dynamical imaging conditions include applications of the critical voltage effect and contrast analyses for identifying faults. These applications are discussed in recent conference reports^(6, 7, 9).

Minerals

The increased penetration capabilities permit HVEM also to be utilized in studies of particulate materials. In fact, current research programs involve characterizing particulate materials ranging from those at the depth of the ocean floor (mineral exploration studies) through pollutants in the earth's atmosphere, terrestrial minerals and dusts and rocks collected from the surface of the moon--truly universal applications.

In the analysis of manganese nodules from the floor of the Pacific Ocean, high resolution HVEM selected area diffraction analyses showed that most particles in the size range 100-540 $\text{\AA}$, of the nodules so far examined contain ferric hydroxide⁽¹¹⁾. Mineral identification from accurate d-spacing measurements is facilitated by computer programs that are also used in environmental research, such as analysis of asbestos fibres which are too thick ($\sim 2\mu\text{m}$) for identification at 100 kV. HVEM also allows these fibres to be examined for longer times because of the reduction in ionization damage at higher energies. Further

examples of mineralogical applications can be found in a recent book⁽¹⁰⁾.

High Resolution Electron Microscopy

In a structural approach to understanding materials at the atomic level, there are four important parameters about which we need information. These are:

- (i) atomic identity;
- (ii) atomic coordination;
- (iii) atomic environment;
- (iv) deviations from perfection.

At present, the resolution of current commercial transmission electron microscopes is limited to $3.5\text{\AA}$ point-to-point and $2\text{\AA}$ line, although as shown in figure 1 atomic resolution in Au has been achieved with a special objective lens with $C_s = 0.7\text{ mm}$. This limit is set by defects in lens systems and mechanical and electronic stability of the instruments. We are not able to achieve atomic resolution and identification in close-packed structures such as metals, although many examples for complex oxides and minerals are now being obtained⁽¹⁰⁾. The aim of a complete atomic description of materials is thus not yet possible. However, with the slightly lower resolution available, lattice fringe imaging is feasible and from these images we can obtain the following information:

- (i) localized crystalline structure;
- (ii) localized chemical composition;
- (iii) configuration and role of lattice defects.

Such information is not generally available using conventional bright and dark field images. It is our experience that the detail revealed by fringe imaging is considerable when compared with conventional imaging modes. It is to be expected then that the logical step to atomic resolution will also open up hitherto unforeseen data.

Figure 5 shows an example at 100 kV for an ordered alloy of Mg_3Cd comparing dark field and lattice images. In the lattice image, atomic steps or ledges at the interfaces are resolved suggesting domain growth occurs by a ledge mechanism. This micrograph clearly shows the precise morphology and distribution of ordered particles in the disordered matrix whereas the dark field images show only the approximate morphology. The main problem is that such images can only be obtained in thin films ($\sim 300\text{\AA}$) so we need HV HREM microscopes in order to study thicker samples.

High Temperature Materials

The investigation of the grain boundaries in silicon nitride is of particular importance at the present time because of the intensive effort in the USA, UK, Germany and Japan to demonstrate that silicon nitride can truly be a structurally important high temperature material, e.g., for turbines, ARPA alone has spent 20 million dollars in five years on this program*. One of the major problems preventing the application of silicon nitride is the rather dramatic decrease in the strength and toughness of the hot pressed form above about 1000 °C. This decrease is generally attributed to the presence of a glassy phase, between the individual silicon nitride grains, which at high temperatures rapidly decreases in viscosity with temperature, flows and allows the grains of silicon nitride to slide past one another. Whilst this model is extremely attractive and the available experimental evidence appears to support the model, no direct observation of a glassy phase between the boundaries has been made until now. We have tackled this problem by imaging directly the crystal lattice planes up to, and on either side of, selected grain boundaries in a 5% MgO hot pressed silicon nitride. Figure 6 shows clearly a non-crystalline phase exists at the triple-boundary junction. The direct imaging method proves that the amorphous phase (probably glass) is very heterogeneously distributed.

*ARPA: Design for brittle solids

Lattice Imaging and Estimating Local Composition

Interfaces

The superior information available from lattice imaging of alloys over conventional imaging modes is illustrated by fig. 7 which compares the lattice image and superlattice dark field image of an antiphase boundary (APB) in ordered Ni_4Mo . In conventional imaging, the translation at the APB must be determined by contrast experiments which can be tedious. However, the lattice image of 420 planes can be taken as representing the positions of the Mo plane of atoms, which occur every fifth plane in the superlattice with the other planes being all Ni. The continuity of the fringes (atomic planes) is shown to within an atomic diameter of the fault. From calculations of the fringe visibility with degree of long range order, it is established that little change in degree of order occurs up to the APB. It can be seen directly that the left-hand side of the boundary is shifted upwards by the two-fifths of the superlattice spacing (i.e., two fundamental lattice planes) with respect to the right and that there is a local change of spacing (and hence composition) where the boundary becomes parallel to the imaging planes (arrowed) where it is non-conservative. The fringes indicate a Ni_5Mo composition in this region. Considerably more information is present in the lattice image which is not available from the conventional micrograph.

The micrograph shown in figure 8 is a lattice image of an alloy which has undergone spinodal decomposition, a phase transformation which proceeds by a homogeneous clustering of like atomic species, in this case gold and nickel, such that a sinusoidal composition variation develops. The modulation is reflected in the large scale image contrast, with light and dark regions representing gold-rich and nickel-rich areas respectively. It was expected that since the difference between lattice parameters of pure Au and pure Ni is $\sim 14\%$, the lattice image (fine fringe contrast in fig. 8) should exhibit

a difference in fringe spacing between light and dark regions. Verification of this effect came from a microdensitometer trace normal to the fringes, a smoothed version of which is shown in figure 9. It was found that measurements of the modulation wavelength (distance between peaks in the composition variation) from both conventional and lattice images agreed exactly⁽¹²⁾.

More accurate measurements can be obtained using laser microdiffraction as shown in Fig. 10 by which changes in lattice parameter of only $\sim 1\%$ can be detected. Such data cannot be obtained by alternative techniques, and illustrates the power of the combination of lattice imaging and optical diffraction.

Since the d spacing is directly proportional to the lattice parameter and composition, it is possible to define the composition-wavelength characteristics to within $10\text{\AA}$. This is to be compared with electron diffraction selected area resolutions of about 1μ (at 100 kV) and STEM of about $30\text{\AA}$ (also at 100 kV). The limit of $10\text{\AA}$ is due to interference from Fraunhofer scattering from the aperture in the optical system, as can be seen in fig. 10.

Applications of this technique currently include studies of grain boundary phase transformations, identification of polytypes in ceramics as well as continued work in ordered and spinodal alloys. Thus, we believe the combination of lattice imaging and optical diffraction will provide such useful information about materials which will be complementary to data obtained by STEM and other analytical methods. Important developments are also expected to occur by HREM studies of amorphous materials⁽¹³⁾.

Current Status and Future Directions

Although we are obtaining considerably more information from lattice images than from conventional micrographs there are still a number of problems preventing us from attaining our goal of atomic resolution and identification.

The resolution of conventional 100 kV instruments is limited. The thickness of specimens for image interpretation in terms of projected change density must be less than $100\text{\AA}$, which is generally not possible for many materials. Both these factors can be overcome by utilizing higher voltage, high resolution instruments. Voltage increase also improves the possibility of studying beam-sensitive materials such as polymers and biological specimens. The interpretation of high resolution images, particularly for the imperfect lattices which are often encountered in practical materials, requires more sophisticated theories than currently used. Image formation is still not experimentally routine and the output of lattice images is low when compared to conventional images. Again, this situation should improve with better instrumentation. Specimen stages for in-situ experiments (e.g., heating, cooling, environmental) are far below even lattice fringe resolution. It is to be expected that much more reliable conclusions can be drawn about materials behavior by actually observing reactions at the atomic level as they occur.

All areas of materials science should profit by atomic resolution, but perhaps the most dramatic development can be in the radiation damage field. To image point defects (interstitials and vacancies), identify the environment in which they exist and observe their migration in various materials would considerably enhance our understanding of this important phenomenon. Such experiments should be feasible in a $2\text{\AA}$ point resolution, high voltage electron microscope, equipped with a high resolution cold stage.

One final point that must be considered is that there is a feeling of excitement in electron microscopy now that we are on the verge of atomic resolution. These results, coupled with the fact that despite the limitations on selected area diffraction due to electron optical errors (spherical aberration) which can be overcome, at least geometrically, by utilizing optical microdiffraction,

indicate unlimited potential for HREM. Further improvements in instrumentation, e.g., beam source, lens aberrations, which will also yield higher diffraction and spectroscopic resolution, can in the final analysis lead to the ultimate goal as iterated in the introduction, viz., atomic resolution and identification.

A new 1.5 MeV microscope is planned for future installation at Lawrence Berkeley Laboratory in 1978. This instrument will be part of a central facility serving many disciplines. With the increasing costs of such facilities the concept of a sharable facility (or center) is receiving more and more attention. This is especially true in the United States where currently all high voltage microscopes are imported. A recent workshop on ultra-high resolution⁽¹⁴⁾ also points to these needs as electron microscopists are now very close to atomic resolution. If this is to be achieved and a HVHREM is to be developed with this capability, such as the 500 kV instrument at Kyoto, and the 600 kV microscope at Cambridge, England, then millions of dollars need to be invested and cooperation between scientists is essential. A major effort is currently underway to try to establish a center for electron microscopy at atomic resolutions at Berkeley. Without such a center, research in this field in the USA must fall yet further behind that of other countries where major programs are already underway (e.g., Japan, England, Germany, and France).

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Figure Captions

- Figure 1. Direct images of gold atoms in a [001] projection of a thin gold film at 10^8 magnification. Courtesy of Professor H. Hashimoto, Osaka University, Japan.
- Figure 2. Scheme showing the information obtainable in a transmission electron microscope.
- Figure 3. a-c shows aging reaction in LiFe_5O_8 . The formation of coherent LiFeO_2 particles (A) improves the magnetic hysteresis properties (curve a). Whilst overaging which causes LiO loss has deleterious effects (curve c). Curve d is as-received. (R. K. Mishra).
- Figure 4. Bright field image of $4\bar{g}$ ($g = \bar{2}20$) at 650 kV with partial dislocations resolved. (b) microdensitometer trace at A (c) 8beam dynamical calculation for the dissociation $1/2[011] = 1/4[011] + 1/4[011]$. (R. K. Mishra).
- Figure 5. Comparison of conventional dark field and lattice images of partly ordered Mg_3Cd . The ordered domains contain the $5.4\text{\AA}$ superlattice periodicity. Unit cell ledges are resolvable at the domain interfaces using lattice imaging.(J.Dutkiewicz)
- Figure 6. Lattice images of Si_3N_4 showing an amorphous region at the junction of these grain boundaries. (D.R. Clarke)
- Figure 7. (a) weak beam dark field image and (b) lattice image across an APB in Ni_4Mo . (R. Sinclair)
- Figure 8. Lattice image of 200 planes in spinodally decomposed Au-Ni alloy. (R. Gronsky) Courtesy Acta Met. (Ref. 12)
- Figure 9. Densitometer trace normal to the fringes shown in Figure 10. (Ref. 12)
- Figure 10. Laser beam optical diffractograms showing directly the modulations in d spacing every $10\text{\AA}$ along [100]. The lower diffractogram is from an area $\sim 200\text{\AA}$ in diameter and shows the satellites or side-bands. (R. Gronsky)

XBB 768-7301

GOLD LATTICE: 150^oÅ THICK FILM, MAG 10⁸X
H. HASHIMOTO. OSAKA UNIVERSITY, AUG. 1976

Fig. 1

XBL 7612-III83

Fig. 2

XBB 766-5125

Fig. 3

XBB 758-5877

Fig. 4

XBB 747-5203

Fig. 5

XBB 769-8516

Fig. 6

XBB 7412-8987

Fig. 7a

XBB 7410-7097

Fig. 7b

XBB 757-5130

Fig. 8

XBL 757-6648

Fig. 9

XBB 762-1583

Fig. 10

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