Title
THE CONTRIBUTION OF ELECTRON MICROSCOPY OF MATERIALS SCIENCE TO SOCIETY

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Publication Date
1987-08-01

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Presented at the Philips Symposium,
Eindhoven, The Netherlands,
September 24, 1987

The Contribution of Electron Microscopy of Materials Science to Society

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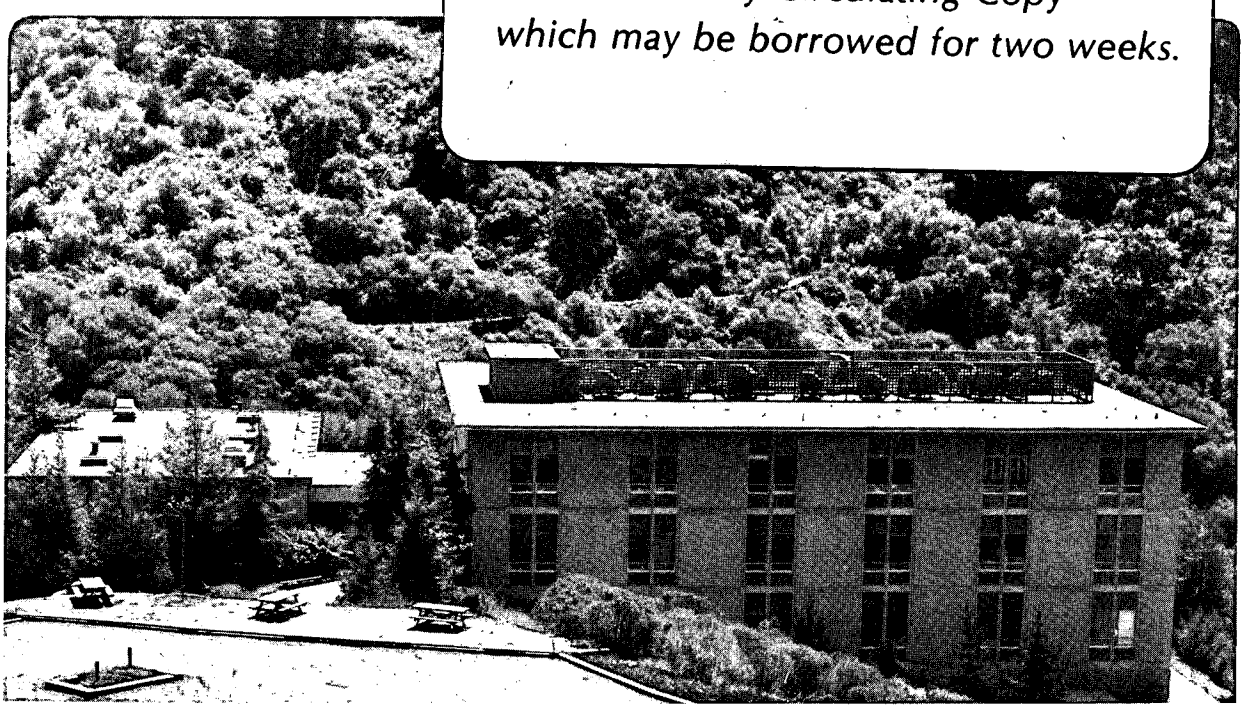
August 1987



Prepared for the U.S. Department of Energy under Contract DE-AC03-76SF00098

DISCLAIMER

THE CONTRIBUTION OF ELECTRON MICROSCOPY

OF

MATERIALS SCIENCE TO SOCIETY

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Philips Symposium in Eindhoven, The Netherlands

to honor Nobel Laureate, Ernst Ruska, Sept. 1987

A. General Introduction

We live and work in a world of materials. Modern technology depends critically on the availability of advanced materials, e.g., in transportation, communication, data processing, production systems, etc. and more and more emphasis is being placed on research and development of materials. The National Academies of Sciences and Engineering in the USA are now completing an intensive study and together with the major federal funding agencies give high priority to materials characterization, especially electron microscopy in spite of its current high costs. Industrial laboratories and even some production facilities, in addition to Universities and leading research centers, have become heavy users of electron microscopes. There are well over 10,000 instruments in use in the Western world. Materials research and development in metals, ceramics, composites, including designing for better mechanical and physical properties, processing, forming, joining, catalysis, etc., all require scanning and transmission electron microscopy because of the scale of relevant microstructure and microanalyses. Economically, electron microscopy represents multibillion dollars per year in acquistions and operating costs, so there is no question about its impact from a business viewpoint.

In addition to the obvious business and economic aspects of electron microscopy production and sales, there are of course enormous intellectual, educational, technological and societal aspects. Because the instrument is central to all fields of structural characterization, electron microscopy is perhaps the most interdisciplinary field in todays complex world. Biologists, materials scientists, engineers, physicists, chemists, etc. all rub shoulders at national and international meetings, such as this one, and, as current President of the International Federation of Electron Microscopy Societies, (IFSEM), it is a great pleasure to see so many national delegates in attendance. The common bond of electron microscopy cuts across political and social boundaries and stimulates international cooperation and understanding. Professor Ruska, whom we are honoring at this Symposium, was the Chairman of the first IFSEM Congress in Berlin (1958), although the first international meeting was held in Delft in 1949.

Since characterisation of structure, microstructure, composition, and properties is an integral part of research and development in the materials sciences, as schematically outlined in Fig. 1, it is a field that is ideally suited for electron microscopy. The primary advantage of electron microscopy, which is truly a diagnostic tool, over other methods of characterising materials is of course its high resolution now at the atomic level, and the ability to detect and record the various events that occur when materials are bombarded with electrons, (elastic, inelastic, coherent and incoherent), so that in one instrument it is possible to combine imaging, diffraction and spectroscopy, in-situ, in both static and dynamic experiments as shown schematically in Fig. 2. The information so obtained provides great insights into materials behavior and helps enormously to understand and improve materials performance. Table 1 summarizes some of these aspects whilst Table 2 outlines some of the main techniques which are utilized by electron microscopists in studies of materials.

From a scientific and technological viewpoint, the impact of electron microscopy on materials science is very substantial and it would be impossible here to properly cover all aspects. Indeed, no attempt will be made to do this. There is no doubt that physical metallurgy, which is concerned with structure-property

relationships in metals and alloys and is the forerunner of materials and condensed matter sciences, developed hand-in-glove with the development of electron microscopy, as one interacted strongly with the other. For example, specimen preparation methods, and instrumental developments were nucleated by the needs of the microscopists. In addition, the development of theory and rapid computational techniques allow interpretation to be easily carried out, now almost instantaneously. Since the electron microscope provides microstructure, diffraction and spectroscopy data at high resolution and in one instrument, (e.g., fig. 2) it is clearly a leading tool in the exploration and diagnosis of the structure of materials. Table 3 outlines some of the main historic developments in materials science research, and illustrates how the growth in techniques, instrumentation, theory and interpretation, have lead to today's remarkable situation where almost any material can be examined even at atomic levels of resolution.

Although the electron microscope was invented in 1931 by Ernst von Ruska and his colleagues, research using this instrument for metallurgy did not start until the 1940's, and then in transmission by using surface replication and extraction replicas (Figs. 3,4). In the 1950's, as a result of improvements in instrumentation and specimen preparation methods, research on internal structure of metals using thin foils, quickly confirmed suspicions and theory that effects below the resolution of light microscopes and not revealed by x-ray diffraction were of great significance, e.g., defects, micro-cracks (Fig. 3) grain boundaries, second phases, etc. The first direct observations of dislocations and stacking faults sharply confirmed the theory of dislocations already proposed two decades earlier. Exciting results were obtained by in-situ experiments on dislocation glide and climb (Fig. 5.) and resulted in particularly intensive research on the plasticity of metal crystals. The more that was learned about microstructure, morphology, defects, crystal structure and local composition (Table 1, Fig. 2)the faster became the application of this knowledge to push the instrument makers to improve instruments, and the materials engineer to improve or even design new materials. Strong, tough, ductile steels designed as microcomposite structures and safe lightweight aluminum alloys that are the basis for today's high performance aircraft and aerospace industries, are typical, representative examples of metallurgical alloy design and development that were a direct consequence of electron microscopy research. Such developments spurred the growth of the instrument market so that the overall impact on technology and society through business and economic growth has been extraordinary. The growth also required increased demands for trained personnel and adequate government funds as the cost of instrumentation has multiplied almost 10 times in the past 30 years. This has lead to the establishment of centralised facilities and National Electron Microscopy Laboratories. State-of-the-Art Electron Microscopy Facilities are now a requirement in any significant university and have made an important impact on education. However, there is still (at least in the USA) a shortage of well trained graduate students in this field.

In the following paragraphs, some examples have been chosen from researches in materials sciences and engineering that have had an important influence on society from a technological viewpoint. As can be seen from Tables 1-3, almost all kinds of materials are now being studied so the examples given below are merely a representative selection and are from the author's personal experiences. A major materials field, viz., semiconductors is reviewed in the papers by Drs. Stacey and Humphries.

B. Some Representative Examples

1. Failure Analysis

Scanning Electron Microscopy has become as indispensible as the light microscope and is now employed for routine failure analyses and product evaluation. Failure analysis initially was carried out largely through replication and scanning microscopy which were used for fractography (Fig. 3). Now failure analysis includes all the tools of electron microscopy and, coupled with fracture mechanics, is itself an established business. Public concern over spectacular accidents, the comet aircraft disasters, bridge failure, ships splitting, even in dock (Fig. 6) - was relieved as a result of solutions provided through such analyses. Nuclear power safety, radiation damage, which can be simulated in a high voltage microscopy (Fig. 7) swelling of materials, and nuclear waste disposal problems, all rely heavily on research using electron microscopy.

Steels are very important in structural applications and very detailed and sophisticated electron microscopy techniques are required to understand them and to design better, more economical alloys. An example is that of quenched and tempered steels which are strengthened by defects introduced by the martensitic transformation on quenching. However, if the alloy content, especially carbon, becomes too high ($C \geq 0.4\%$) the morphology of the transformation product, martensite, changes from packets of dislocated laths to twinned plates, and fracture toughness at the same strength level, drops. These results quickly showed that fracture toughness, as strength, is a microstructure sensitive property. This point is illustrated in Fig. 8 obtained during development work on steels for landing gear on heavy aircraft. Such research was stimulated by frequent landing gear failures in the early days of jet aircraft. Figure 9 shows from fracture mechanics, how the toughness parameter K_{IC} determines the critical crack size-stress relationships for crack propagation. These concepts in combination with extensive observations by electron microscopy research has lead to the development of much safer steels. A similar problem existed with the Al-Zn-Mg type aluminum alloys used in the first "Comet" jet aircraft which were designed from strength rather than toughness considerations. Tragic crashes occurred as a result of using alloys with inhomogeneous microstructures and of low toughness, especially due to the "precipitate-free zones" (PFZ) adjacent to grain boundaries which lead to intergranular failure. It was electron microscopy which ascertained these microstructures and which helped lead changes in alloy design, alloy composition, and processing. Today's jet aircraft are probably the safest form of public transportation.

Figure 10 is an illustration of the way in which various techniques must be combined to synthesize materials. This example is for the analysis of low or medium carbon martensitic steels which have been designed to develop microcomposite structures consisting of fine packets of lath martensite and untransformed austenite. Since carbon is almost impossible to analyse spectroscopically in steel foils it took the combined efforts of several imaging and analytical methods and eventually proof by atomic spectroscopy using a field ion atom probe instrument to confirm that partitioning of carbon between austenite and martensite is a key factor in the stabilization (and hence, mechanical properties) of the austenite.

2. Light Alloys: Modern Developments

One of the few "conventional" physical metallurgical areas currently receiving intensive and broad support is that of Al-Li base alloy development. For these alloys, the advantages of weight savings and concommitant fuel savings is considerable (perhaps up to 10% for a typical commercial aircraft). The problems with these alloys in some ways resemble those of the Al-Zn-Mg alloys developed in the early 1950's such as the formation of non-homogeneous microstructures and hence very anisotropic properties. One of the basic phases in precipitation strengthening of the Al-Li base alloys is the δ' phase (Al_3Li) which is often associated with the β' phase (Al_3Zr). Atomic resolution imaging of this phase and image computation (Figs. 11, 12) show that the structure is ordered with little or no strain at the δ'- β'interface. The change in contrast (fig. 11) at the interface is basically due to the change in composition (and hence the projected potential). Research on such Al-Li base alloys is currently being actively pursued almost world-wide in the race to produce economical, and safe, lighter alloys, and electron microscopy is playing an important role in this effort. Lithium, due to its low atomic number, poses great difficulties for spectroscopy, but special diffraction effects available in high voltage instruments, viz., the critical voltage effect is already proving to be an unique technique for helping to solve this problem.

3. Non-metallic Inorganic Materials

The expanding interest in "new" or so called "advanced" materials is resulting in a gradual decrease in research and development in structural metallic systems. However, there is concommitantly increased activity in work on many other materials, e.g., ceramics and composites. Indeed, the enrollment in metallurgy programs at major universities is declining to sometimes alarming proportions.

Progress in applying microscopy to non-metallurgical materials such as ceramics was considerably spurred on by the Apollo space missions, which involved electron microscopy studies of lunar and terrestrial minerals, developments in TEM instrumentation and specimen preparation techniques, Table 3. The impact of electron microscopy in ceramics is especially notable in understanding structures produced by various processing routes and hence their subsequent properties. Ceramics are generally produced from small powders and often require additives to achieve densification. It was electron microscopy which first proved the existence of amorphous phases often only one or two molecular layers thick at the grain boundaries after sintering, hot pressing, etc. An example is shown in Fig. 13. The problem is thus a generic one in ceramics and applies to many systems as is shown in Fig. 14. Imaging and spectroscopy of the grain boundary regions has been instrumental in recognizing new or improved methods of processing which eliminate or minimise the deleterious effects of these structures. In addition, the development of energy loss spectroscopy which is particularly useful for light elements can also indicate the bonding character and form of an element in the material. Figure 15 is an example for SiC bonded with excess carbon forming at pores as graphite.

A further illustration of the importance of electron microscopy in ceramics is shown in figs. 16 a-d for Mn-Zn ferrites which is still an important material in magnetic recording. The ability to recognize calcium segregation (added for resistivity) which causes magnetostriction (as shown by the distortion of the CBD pattern) and loss in permeability in conjunction with the lorentz imaging, which directly shows magnetic domain wall pinning at the boundaries, has lead to

developments which eliminate this problem. This example is typical of applications in an industry which is growing rapidly and amounts to several billions of dollars per year. An understanding of problems associated with boundaries in ceramics in areas such as creep performance of ceramics for new gas turbine engines, fracture resistance, ionic conductivity (e.g., beta sodium aluminate), magnetic permeability (soft ferrites) and electrical conduction, (e.g., warm superconductors) are but a few examples of current resarch and development efforts in ceramics. In the present decade, ceramics research seems now to be replacing physical metallurgical research as one of the most active fields of electron microscopy applications. The same can be said of semiconductor materials but these are covered elsewhere in this Symposium.

Another area which has become extremely active and important is that of toughening of ceramics for structural applications utilizing several principles learned from metallurgy. One of these draws from metallurgical experience of martensitic transformations, so important in ferrous alloys, especially using ceramic alloys with zirconia. Zirconia ceramics can be stabilized or partially stabilized by alloying with other oxides (i.e., which lower the martensitic transformation temperature). Under stress, the zirconia can transform rather than crack, and such systems are said to be "transformation toughened". In-Situ HVEM has in fact demonstrated this process very beautifully. Further developments include ceramic composites where zirconia is dispersed in a compatible matrix, e.g., mullite. In these cases several toughening mechanisms may apply, such as microcracking and crack path deflection, as has been demonstrated by electron microscopy (scanning and transmission).

4. Asbestos: National Policy and Public Health

Asbestos refers to silicate minerals which can exist in many forms and hence have different properties and has now been recognized as a severe health hazard contributing to lung diseases. Asbestos fibers are used as insulating materials in buildings, brake linings in automobiles, etc. and fine fibers of this material, often not detectable by light microscopy, escape into the atmosphere and can be inhaled and trapped in the lungs. Figure 17 is an example where a HVEM was used to analyse air samples from a California freeway. The fiber has recrystallised as a consequence of the frictional heat developed during braking and the resulting surface "roughness" may contribute to the "sticking factor" when these fibers are inhaled in the lung.

For many years, assessments of exposure to asbestos fibers were based on polarized light optical microscopy and were limited to fibers more than 10 microns in length. Levels of 5 fibers/cc of air were considered to be acceptable. More recently the likelihood of adverse health effects due to exposure to asbestos has been extended to include fibers as small as 0.5 um in length and only 0.1 um in diameter, i.e., well below the limit of the optical microscope.

Electron Microscopy has played a major role in first uncovering the existence of substantial concentrations of sub-micron asbestos fibers, then relating their dimensions and structures to health effects. Electron microscopy and diffraction is now designated as the analytical method to be used to certify that public schools are free of hazardous asbestos. For example, positive identification of fiber species using electron diffraction and x-ray spectrocsopy is now specified in new U S. Government regulations (See EPA proposal rule, Federal Resigter April 30, 1987, p. 15820-15882). It is probable that the provisions of AHERA (Asbestos

Hazard Emergency Response Act) will be extended to apply to all public buildings and places of employment.

5. An Exciting New Era - Warm Superconductors

The discovery just a year ago of superconductivity above 90°K in copper oxide ceramics containing rare earths has been a major stimulus to ceramics and condensed matter sciences because the ability to achieve superconductivity at using liquid nitrogen, rather than helium opens up enormous economic possibilities for great changes in technology. The optimum material so far seems to be the orthorhombic $YBa_2Cu_2O_7$ compound. However, there are problems which are structure related. One problem is that the critical currents are too low ($<10^4 A/cm^3$) to be maintained and this is almost certainly determined by grain boundary structure and composition in addition to the transformation (tetragonal-orthorhombic) induced microtwinning that occurs. Thus, the problems of polycrystalline superconductors are similar to those generally discussed above for ceramics (fig. 14) and intensive efforts are being made to understand the processing-structure-property relations in these materials. An interdisciplinary effort at the University of California at Berkeley includes atomic resolution imaging wherein the structure and faults can be "fingerprinted" as shown in fig. 18. Well prepared samples do not show intergranular phases (fig. 19) but the large changes in orientation could contribute to the reduced Meisner effect.

A further problem with these materials is their low stability. It has been shown that decomposition can occur in air, or vacuum by diffusion in the a-b planes (fig. 18) with amorphitization at the surface. Thus c-axis orientation control to produce single crystals would appear to be the most promising approach at this time (for applications in supercomputers). Although the potential economic impact on power transmission, levitated transportation and the like is clearly enormous, the problems are basically those for materials sciences, i.e., processing and microstructural control. At this point these problems appear to be very formidable indeed.

C. Summary

The above examples are just a few of very many that could be used to illustrate the impact of electron microscopy in materials upon society. Electron microscopy, materials science and engineering represent classic examples of demanding and interdisciplinary team efforts. Whilst in its early days, electron microscopy was mainly used as a sort of forensic tool to understand existing problems it is now becoming more and more used in the development and design of new or improved materials required for advanced systems - to conserve energy and materials. Over a century ago, H.C. Sorby was the first scientist to develop the use of thin sections of minerals for optical microscopy. Today we use 1000 times thinner sections in order to resolve the atomic arrangments in powerful electron microscopes, at the surfaces, interfaces and inside crystal grains. Materials science and engineering cannot progress without the electron microscope and it is a delight and a privilege to be deeply involved in this exciting area.

Acknowledgements

It is an honor an pleasure to dedicate this brief review to Professor Ernst Ruska who gave us the instrument in the first place, - over 55 years ago - and to whom the Nobel Prize was finally awarded last year.

This work is supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Materials Sciences Division of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098. Structural ceramics research was supported by the National Science Foundation under Grant DMR-80-23461.

I am very grateful to Dr. R.M. Fisher for constructive input to this paper and especially in providing information on the asbestos issues.

Figure Captions

Fig. 1. Schematic of iterative relationship between materials processing, structure and properties and the importance of characterisation.

Fig. 2. Schematic showing the information available from a modern electron microscope in terms of high resolution images, diffraction patterns (CBD) and spectroscopic (shown is an ED X-ray spectrum of a glass standard). The main aspects receiving attention now are development of field emission guns, specimen preparation, in-situ (damage), parallel detection EELS and medium voltages (300-500 Kv).

Fig. 3. Scanning electron micrographs showing ductile fracture initiating at inclusions, in mild steel, and below and surface oxide replica of Al-7% Mg alloy showing sliplines and fracture of an intermetallic phase.

Fig. 4. Transmission electron micrographs from a thin foil of an alloy steel compared to carbon extinction replica (top right) and the EDS x-ray analyses (bottom right). Note that many small particles may not be extracted in the replica. The carbides are identified by selected area diffraction.

Fig. 5. In-situ isothermal annealing experiments showing growth of helical dislocations in a quenched Al-Mg alloy by dislocation climb. Such experiments yield data on diffusion and solute atom-vacancy binding energies.

Fig. 6. Brittle fracture of a ship (see fig. 8)

Fig. 7. Electron radiation damage in a Ni/Ti alloy at 1500 KV (time sequence from A-D is 0-15) minutes causes amorphitization of the alloy as evidenced by loss of Bragg contours and diffuse ring replacing Bragg relections (E to F).

Fig. 8. Showing the relationship between the fracture toughness (crack propagation resistance) on microstructure for martensitic steels, as a function of yield strength.

Fig. 9. Plot of critical crack size for spontaneous crack propagation as a functionof applied stress for three similar steels - the upper one being specifically designed for high K_{IC} toughness. Notice that low K_{IC} materials will crack easily at stresses well below the yield strength (cf fig. 6).

Fig. 10. Characterisation of microcomposite martensite - retained austenite steels.

Fig. 11. Atomic resolution image of Al/Li/Cu/Zr alloy aged to form ordered δ' phase which surrounds β' and matrix M. The superlattice structure of both phases is well resolved (ARM image at 1 mev).

Fig. 12. Multislice computer simulation of the structure in Fig. 11 (Scherzer defocus 180A^{o} thick foil at 1 mev).

Fig. 13. Bright field, diffuse dark field and EDS analysis of sintered Si_3N_4 showing amorphous intergranular phase containing Ca, Si, Al, Mg. Such phases greatly lower creep resistance.

Fig. 14. Schematic diagram showing generic microstructures in processed ceramics and some properties related to these.

Fig. 15. Electron energy loss spectroscopy of SiC sintered with excess carbon a) bright field image b) EELS spectrum from carbon region, c)d)e) standard EELS spectra from amorphous, graphitic and diamond samples. (Courtesy B. Dalgleish).

Fig. 16. a) Diffuse dark field images of MnZn ferrite doped with CaO showing amorphous intergranular grains.
b) EDS analysis showing Ca distribution leading to large strains
c) CBD patterns showing reduction of symmetry from 4 to 2 fold due to strain for patterns taken adjacent to the boundary and far from the grain boundary
d) Lorentz imaging in the under (left) and over (right) focussed condition showing pinning of domain walls at grain boundaries. The central figures are schematics to illustrate these interactions.

Fig. 17. Asbestos fibers imaged at 650 KV showing the recrystallization occurring after braking in an automobile. Such fibers exist in the atmosphere and are harmful.

Fig. 18. Atomic resolution image of the orthorhombic 1-2-3 $YBa_2Cu_3O_7$ in [010] projection a) digitized image b) averaged over 10 unit cells parallel to the fault plane c) rotation averaged after b, d) multislice "semper" program calculated image e) projection of the model used for the 1-2-3 phase and f) the 3-D presentation (arrowed) of the faulted rod structure. The (ab) fault plane corresponds to the plane of decomposition of the compound when exposed to the environment.

Fig. 19. Grain boundary in tetragonal 1-2-3 superconductor with grains in [100] and [441]. There is no intergranular phase.

TABLE 1

MATERIALS	EXAMPLES OF PHENOMENA	INFORMATION FROM EM
Metals Semiconductors Ceramics Minerals Polymers Glassy composites	Defects Radiation damage Phase transitions Implantation Interfaces and Surfaces Small particles Catalysts Pollutants Quasi-crystals Superconductivity Structure sensitive properties	Diagnostic : Crystallography crystal structure atomic and lattice imaging, microstructure morphology, composition distribution, bonding ; Dynamic in-situ studies; kinetics ; Relation to properties (mechanical, chemical,etc)

TABLE 2

Electron Microscopy Characterization

Subjects	Techniques	Information
Microstructure	Imaging:	defects
	amplitude contrast	shape and size distribution of components
	phase contrast	lattice and atomic imaging surface and interface structure
	Selected Area Diffraction	crystal structure and preferred
	Convergent Beam Diffraction	orientation, localized information
Kinetic and morphology	Dynamic, In-situ studies	phase transitions, nucleation and growth, etc.
Microchemistry	Energy Dispersive Spectroscopy	highly localized chemical
	Electron Energy Loss Spectroscopy	information
	Alchemi	atom sites
	Diffraction (CBD, Kikuchi)	lattice parameter--composition
	Lattice imaging	
Magnetic structure	Lorentz microscopy	magnetic domains and
	Differential Phase Contrast	walls

Advantages: Non-averaged information (cf. X-rays, neutrons)

TABLE 3

Outline of Developments of TEM in Materials Science Research

Year	Specimens	Applications/Developments	Instrumentation	Resolution[+]
1932—First operational transmission electron microscope, Berlin (Ruska, Knoll)				
1938—First commercial TEM, Berlin (Ruska, von Borries)				
1940/50	Replicas 1. Oxide 2. Carbon 3. Plastics	Surfaces Slip steps Extracted particles Fractography	50kV instruments Single condenser Little or no theory	~100Å 1946 Hiller
1949—Heidenreich published first paper on TEM of tin foils; basic theory outlined				
1950/60	Thin foil techniques 1. From bulk 2. Deposited	Defects Phase transitions	100kV instruments, contrast theory developed	~ 5Å-20Å
Many developments in instrumentation, specimen preparation methods, and image contrast and diffraction theory for interpretation of data				
1960/70	Metals Non-metals, semiconductors Ceramics (ion thinning) Minerals	Dynamic, in-situ studies: Information explosion on substructure of solids Radiation damage Microdiffraction	First HVEM built in Toulouse (1.2MeV); first 3MeV HVEM built in Toulouse; accessories for in-situ studies; controlled experiments	3Å
1970/80	As above Catalysts	Theories for high resolution interpretation developed Quasicrystals	TEM/STEM analytical, convergent beam; spectroscopy EDXS, EELS commercial HVEMs 0.5-1.5MeV; general acceptance	2Å
Structure imaging to ~2Å interpretable resolution; lattice imaging widely used				
1980/90	Virtually all materials	Atomic resolution in close-packed solids; surface imaging, small particles; fast computation facilities for interpretation, simulation	Medium voltage HREM/AEM(100-400eV commercially available; improved analytical capabilities; parallel detection in EELS wide-spread applications in all fields; UHV microscopes; tunnelling microscopes	1.5Å
1986 Nobel Prize to Ernst von Ruska and for tunnelling microscope (Binnig and Rohrer)				

[+] Interpretable point-to-point

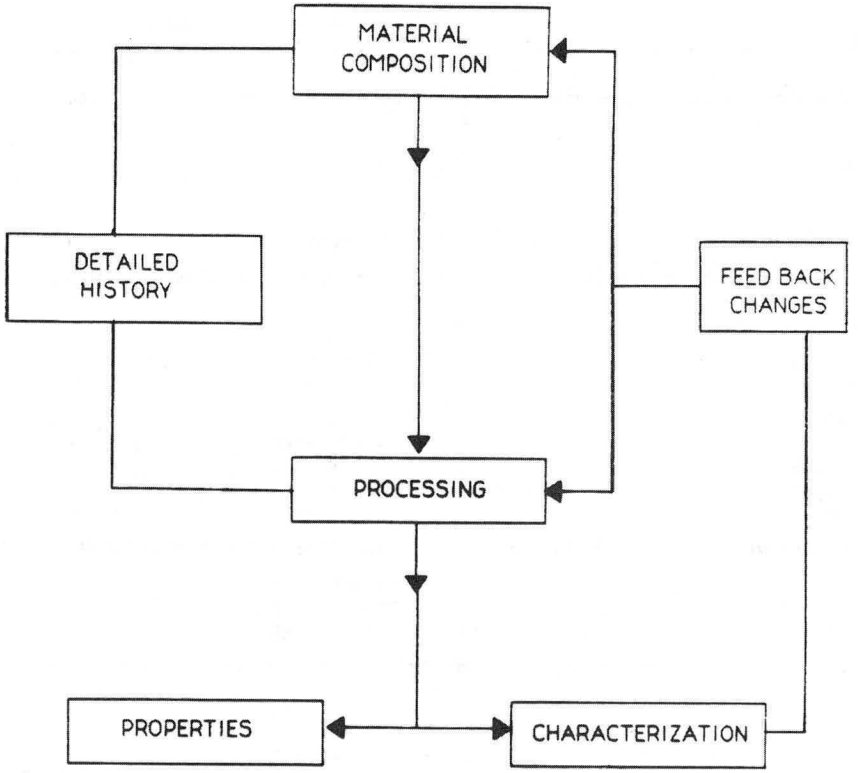

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Fig. 1

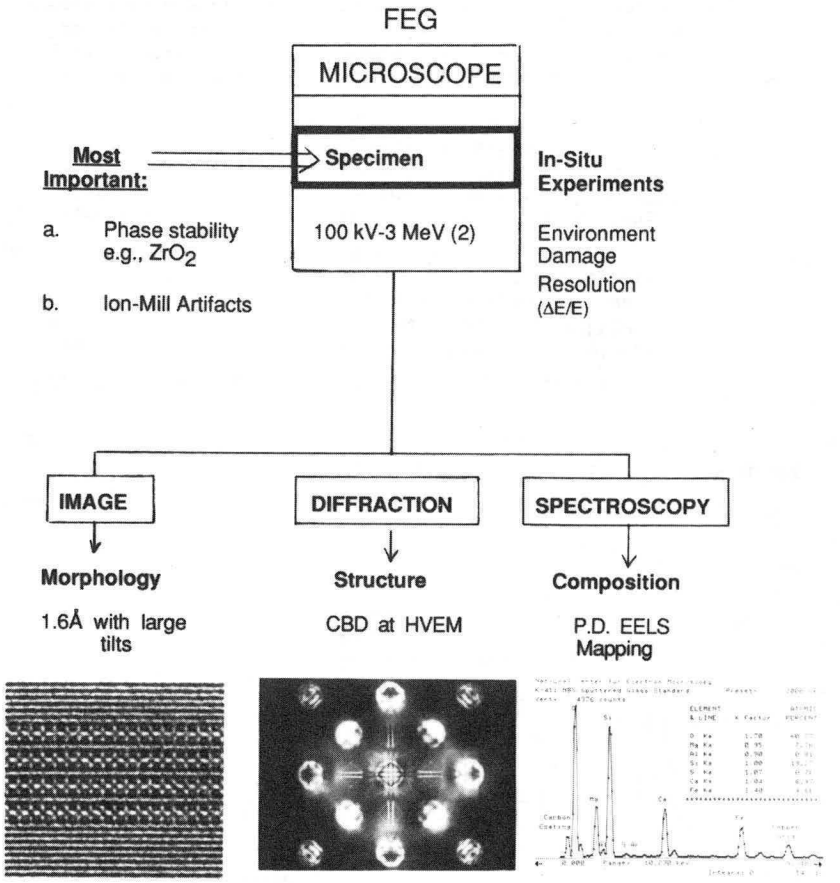

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Fig. 2

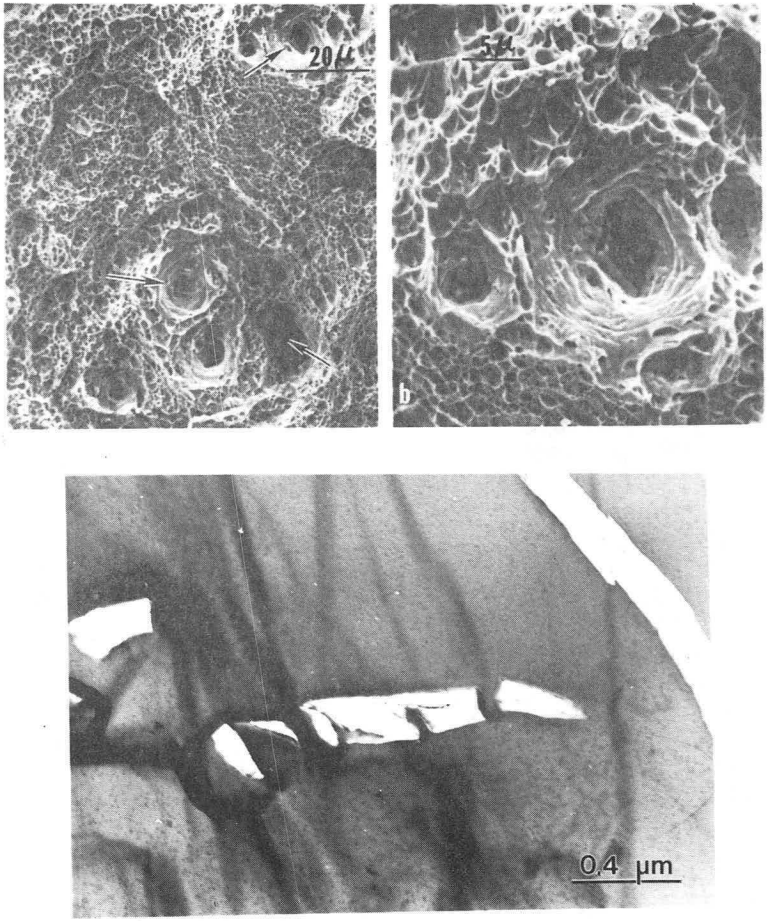

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Fig. 3

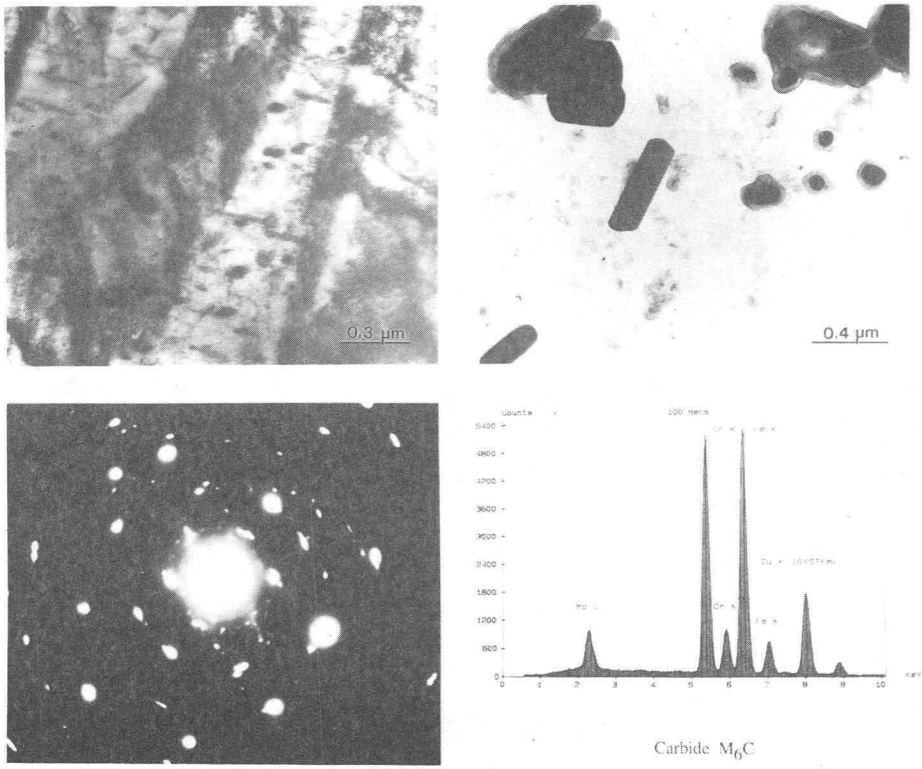

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Fig. 4

DIRECT OBSERVATION OF THE GROWTH OF PRISMATIC
DISLOCATION LOOPS AND HELICOIDAL DISLOCATIONS

Direct observation of the growth of prismatic dislocation loops (e.g. A) and helicoidal dislocations (e.g. B) during an in-microscope isothermal annealing experiment. The material is an Al-5 wt.% Mg alloy which has been quenched from 550°C and aged at room temperature for about 5 years. The isothermal annealing temperature was 130°C. The diffraction conditions were kept nearly constant throughout the experiment by employing a Valdre double-tilt hot stage.

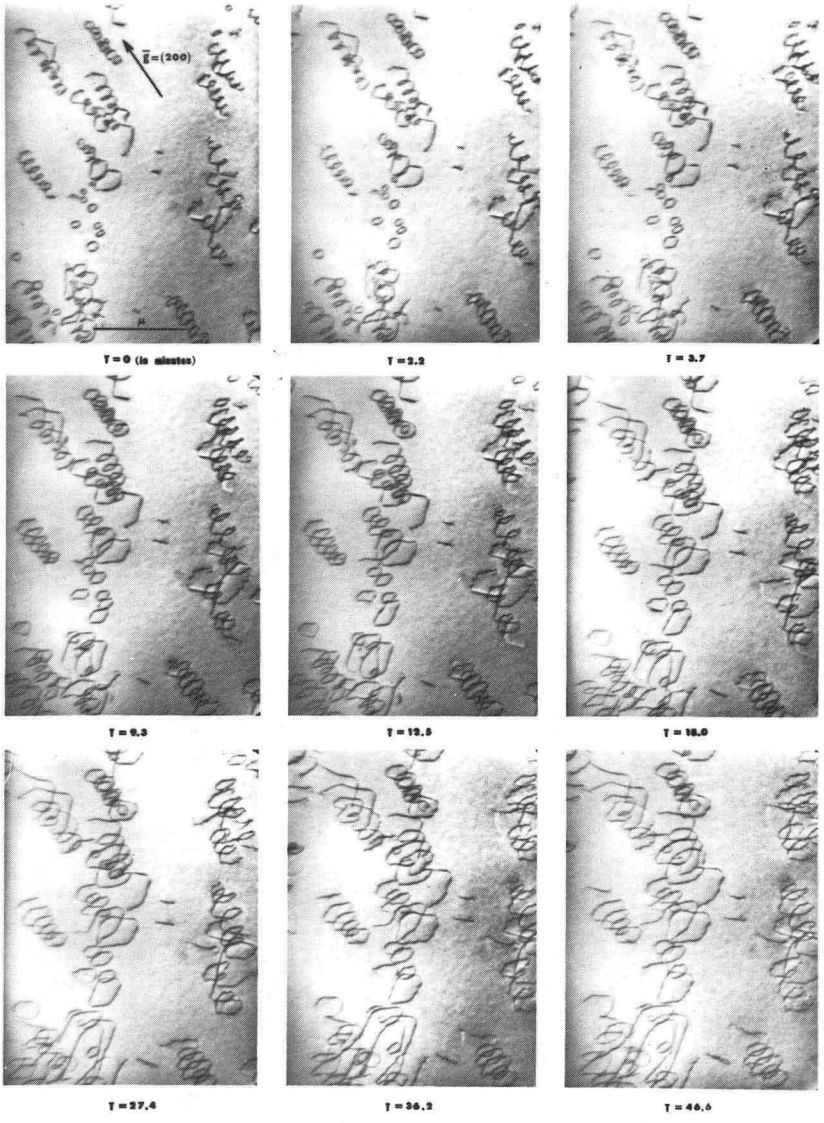

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Fig. 5

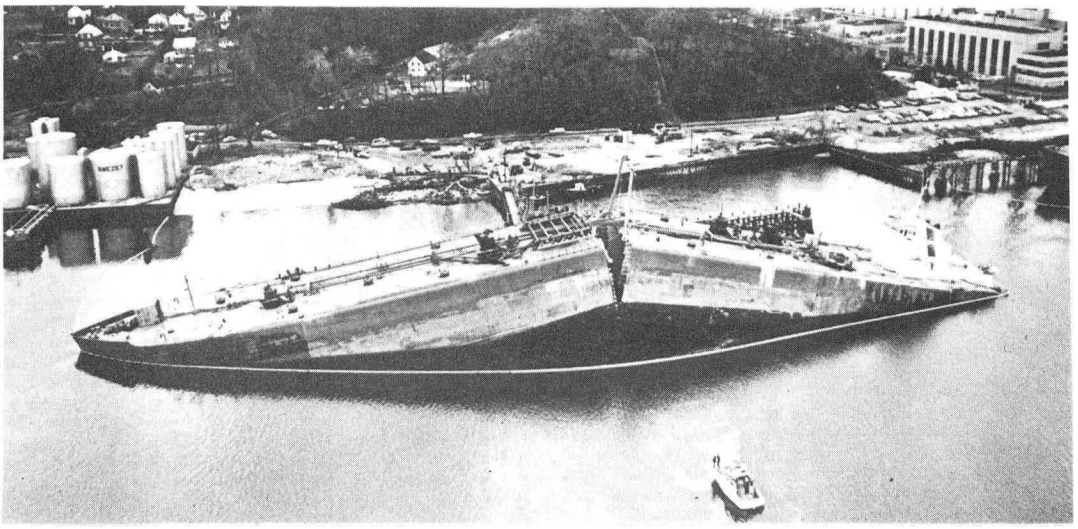

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Fig. 6

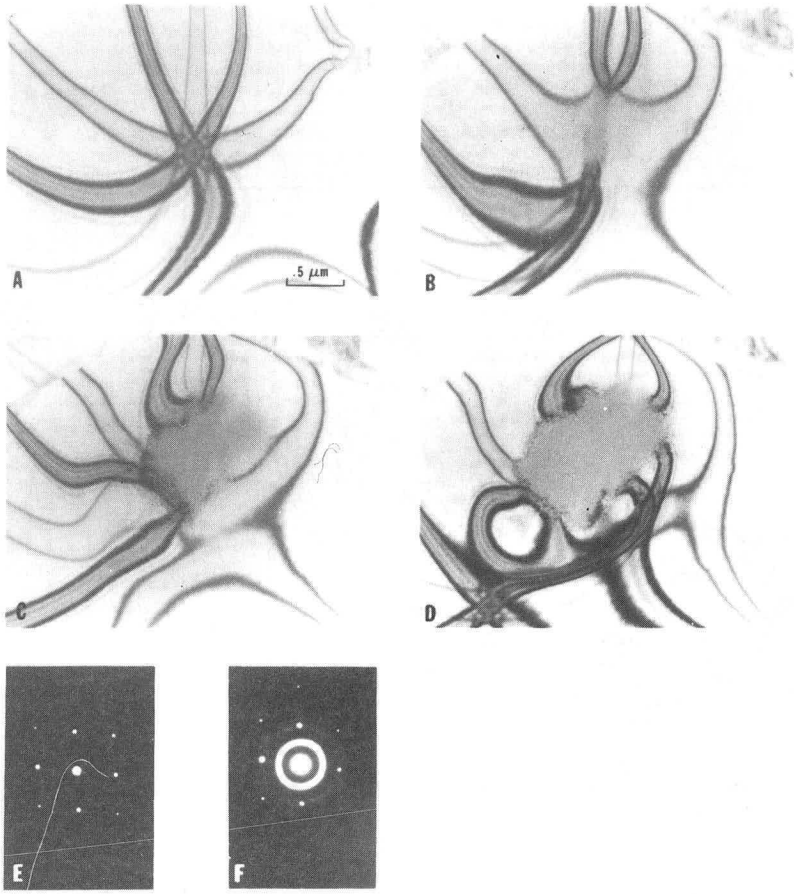

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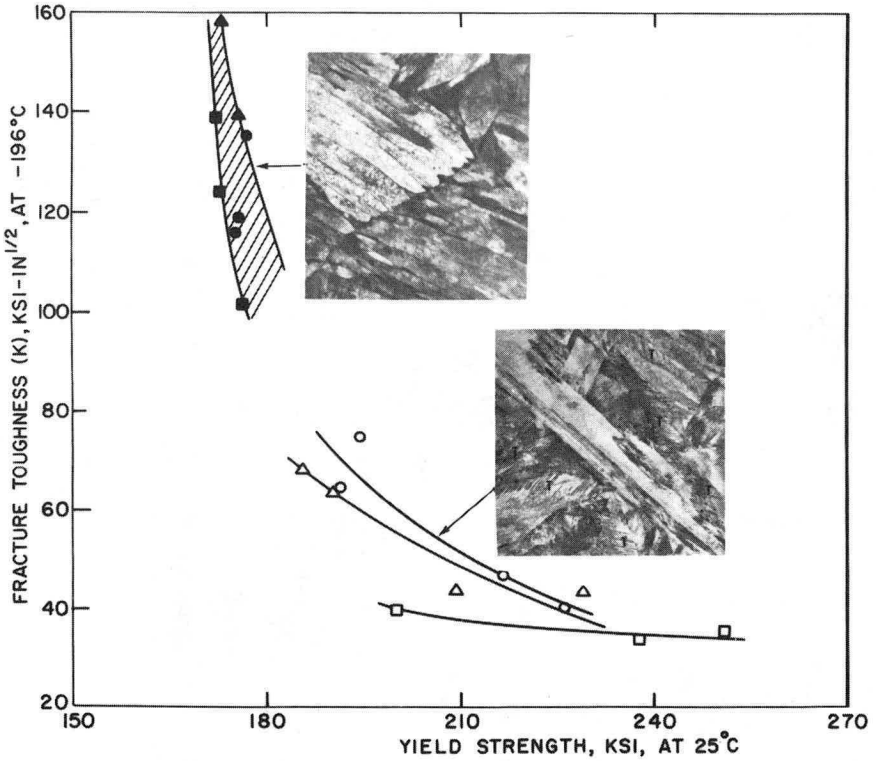
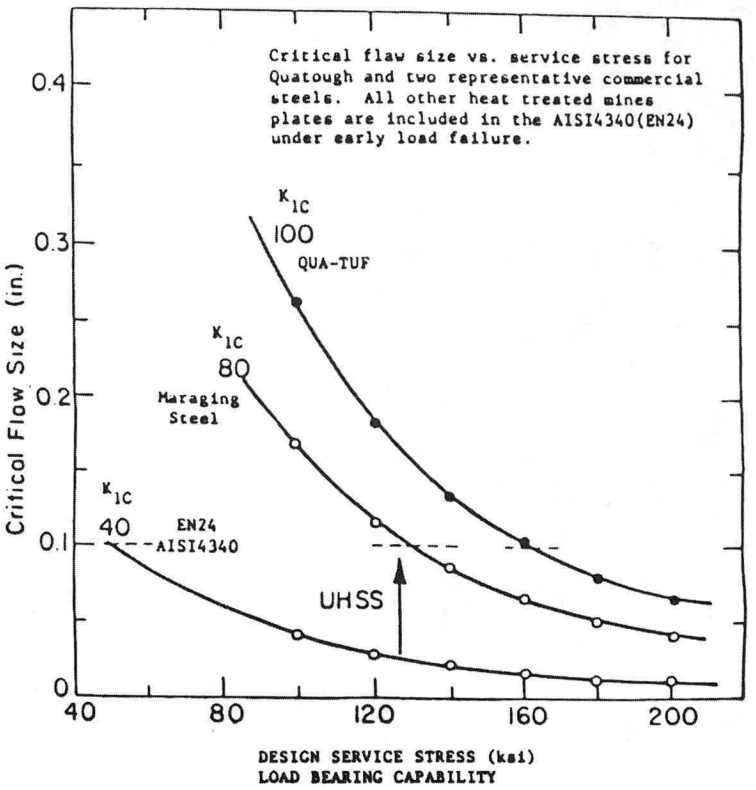
Fig. 7



XBB 701-475

Fig. 8



XBL 8510-4149

Fig. 9

CARBON PARTITIONING IN RETAINED-AUSTENITE IN LATH-MARTENSITIC STEELS

TRANSMISSION ELECTRON MICROSCOPE - IMAGE STUDIES

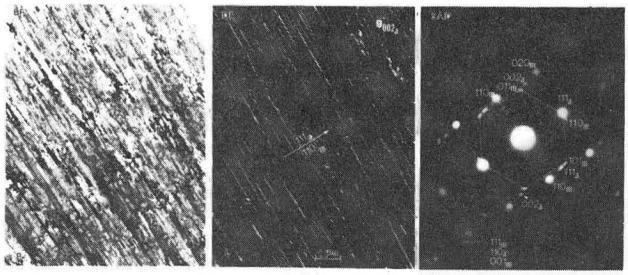


FIGURE 1 - (a) BF and (b) DF (002_γ) images illustrating the general appearance of thin films of Ret-γ at the martensite lath boundaries (0.5 at.% C steel). (c) Composite SAD pattern of martensite (m) and austenite (a).

The microstructure consists of dislocated lath martensite with fairly straignt boundaries and thin film Ret-γ at the lath-like martensite crystal boundaries. DF micrographs (fig. 1) shows an extensive amount of Ret-γ (5 vol.%) even at this low carbon level. The existence of this high temperature phase at low temperatures is attributed to several mechanisms in which interstitial C stabilizes the austenite. (i) Chemical stabilization: Diffusion and partitioning of C in Ret-γ decrease the local M_s temperature and inhibit further transformation. (ii) Thermal stabilization: During quenching interstitial C forms dislocation atmospheres in α' and at the α'/γ interface, pinning the dislocations and suppressing interface motion. (iii) Mechanical stabilization: Part of the austenite to martensite shear transformation strains is accommodated by soft γ which deforms extensively to prohibit the transformation.

The average C concentration in Ret-γ can be determined by measurements of shift in positions of the holz lines in CBED patterns in relation to the change in the lattice parameter of the Ret-γ, due to C: $(\Delta a_\gamma/a_{ref}) = (2/3)[(p_\gamma/q_\gamma) - (p_{ref}/q_{ref})](8/115)^{\frac{1}{2}}$. Ni (99.99% with $a_0 = 3.5238$) was used as a reference, and results cross-checked with Cu (99.999% with $a_0 = 3.6150$). For the example shown in fig. 2, C at.% = 4.9 ± 0.6 (at.% C alloy = 0.7) taking $a_{Ret\text{-}\gamma} = 3.555 + 0.044x$ (w/o C).

Retained austenite (Ret-γ) has been identified in a number of carbon containing lath martensitic steels with M_s and M_f temperatures well above room temperature. Because of its beneficial effects on the mechanical properties (especially fracture toughness) of HSLA steels the influence of interstitial C in stabilizing the γ has been studied in detail using TEM, CBED and FIM-APA techniques.

Steels were austenitized at 1100°C, and oil quenched. TEM foils were prepared by electropolishing in chromacetic at room temperature (RT), and Cu and Ni standards in 25% NHO_3 in CH_3-OH at -30°C. Some steel foils were etched for CBED by "dipping" in 15% $HClO_4$-5% $C_3H_5(OH)_3$-CH_3COOH at -25°C. FIM tips were electropolished in 25% $HClO_4$ in CH_3-COOH at RT.

CONVERGENT BEAM ELECTRON DIFFRACTION

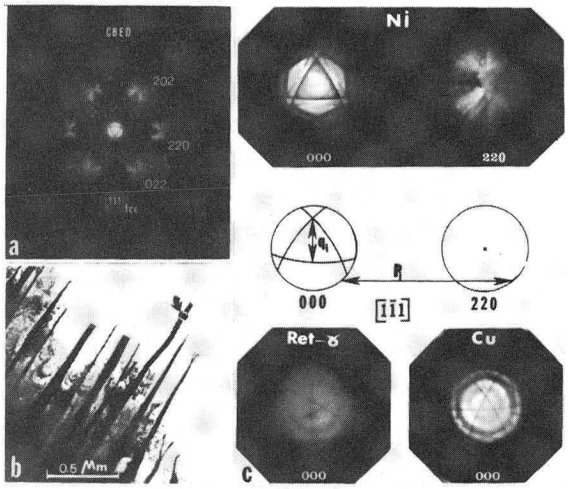
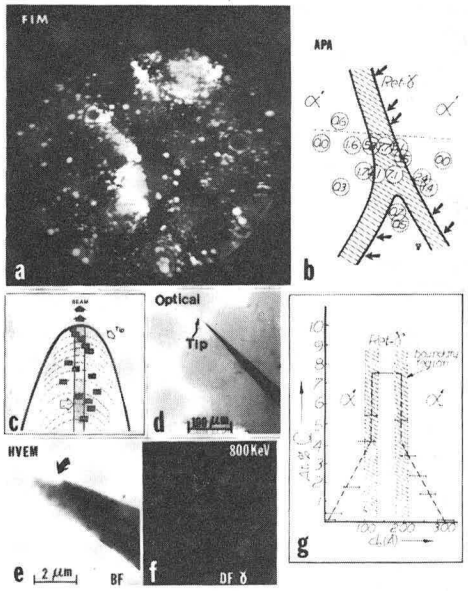


FIGURE 2 - (a) Nickel (fcc) 111 CBED-pattern at 100 kV reveals trigonal symmetry. (b) BF image, from a specially prepared foil, shows the Ret-γ films extending into the perforation. (c) Discs formed by forward scattered beams from Ni, Ret-γ, and Cu (note q_1 and P_1).

FIELD ION MICROSCOPY - ATOM PROBE ANALYSIS



Atom probe analysis provided direct quantitative determination of the C distribution in α' and Ret-γ at 20-30 Å resolution. Considerable C enrichment occurs in Ret-γ - direct evidence of chemical stabilization (figs. 3-4). Detailed measurements of C distribution in a thicker Ret-γ film (fig. 4) gave an average concentration of 3.0 at.% and up to 8.5 at.% at the α'/Ret-γ interface (Thermal stabilization). The extent of deformation (Mechanical stabilization) is discernible in TEM micrographs in fig. 4. There is no apparent change in distribution of substitutional alloying elements (Cr and Mn, fig. 4) in α' and Ret-γ. Hence changes in the amount of Ret-γ with alloying elements are attributed to their interaction with C influencing its mobility. Thus the overall stability of thin film Ret-γ is due to effects of several mechanisms.

FIGURE 3 - (a) FIM image of Ret-γ. (b) Regions of APA analysis. (c) Illustration of the analysis of subsurface regions by field evaporation. (d) Low magnification image of the tip. (e) HVEM BF and (f) DF images reveal Ret-γ. (h) Concentration profile for C across the interface.

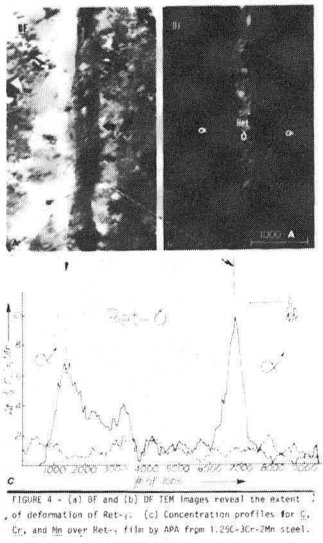
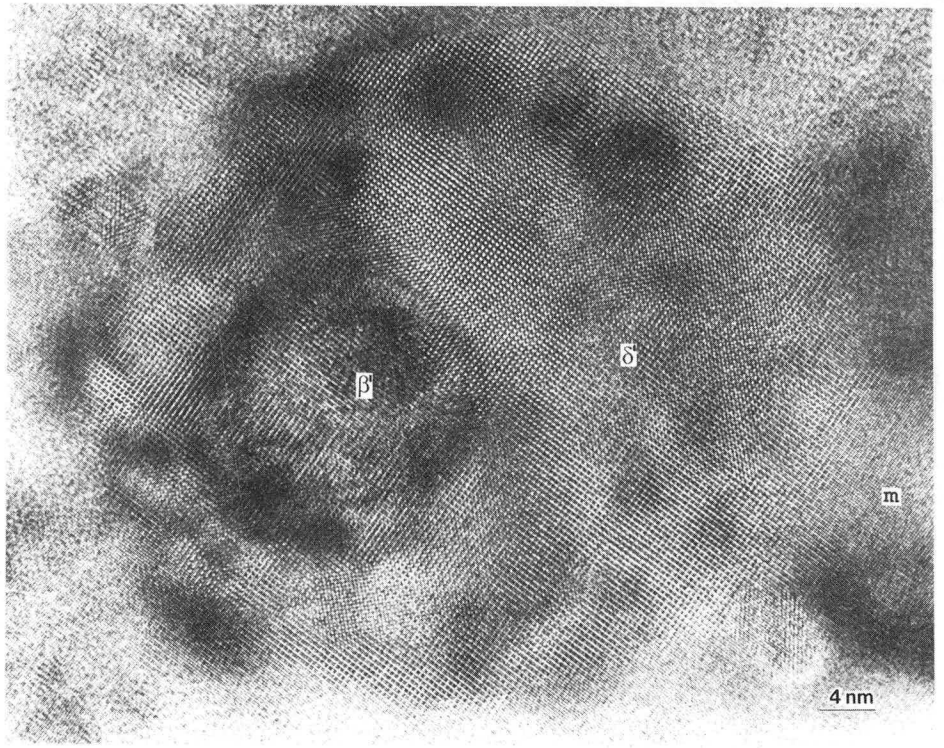


FIGURE 4 - (a) BF and (b) DF TEM images reveal the extent of deformation of Ret-γ. (c) Concentration profiles for C, Cr, and Mn over Ret-γ film by APA from 1.25C-3Cr-2Mn steel.

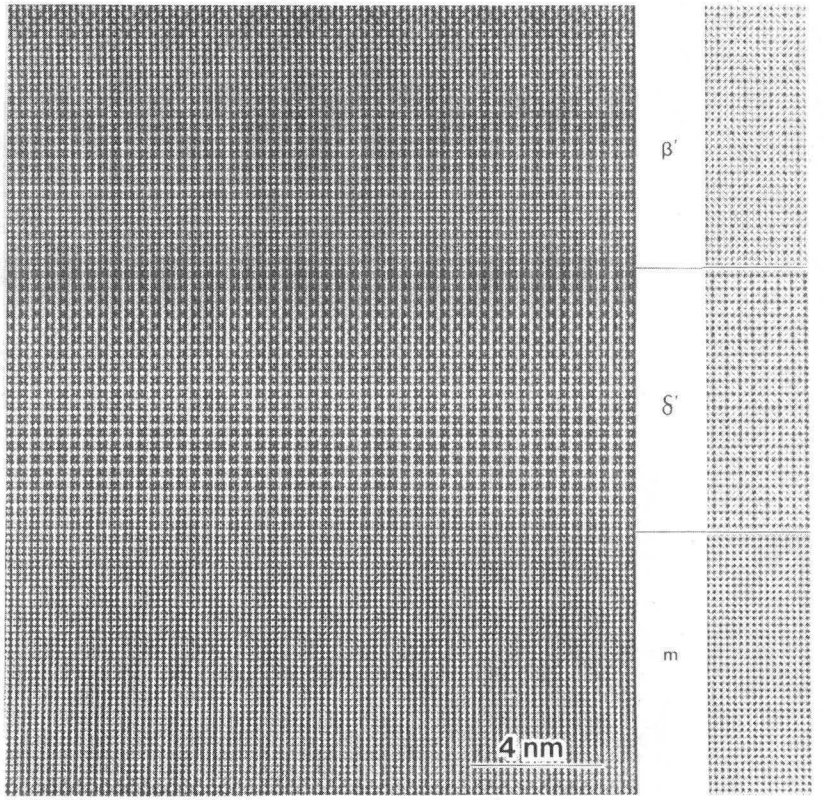
XBB 817-7029

Fig. 10



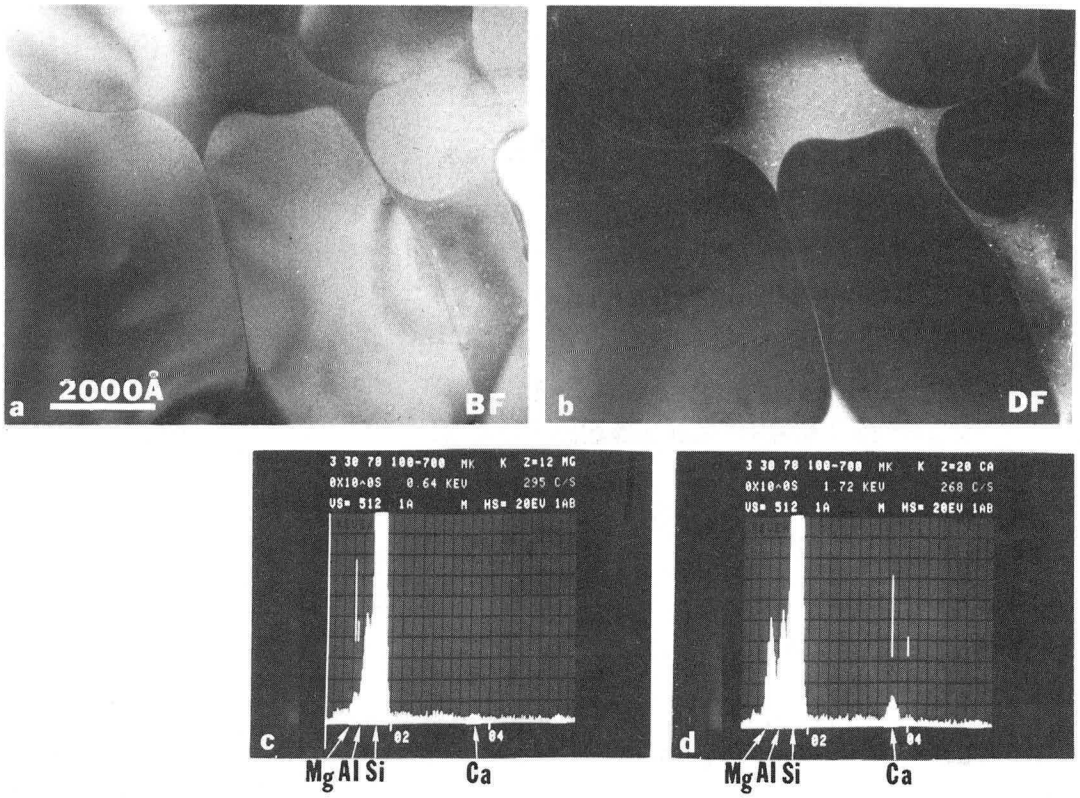
XBB 875-3898

Fig. 11



XBB 875-3897

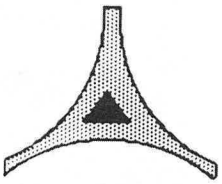
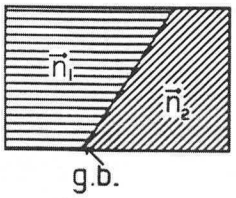
Fig. 12



XBB 785-5933

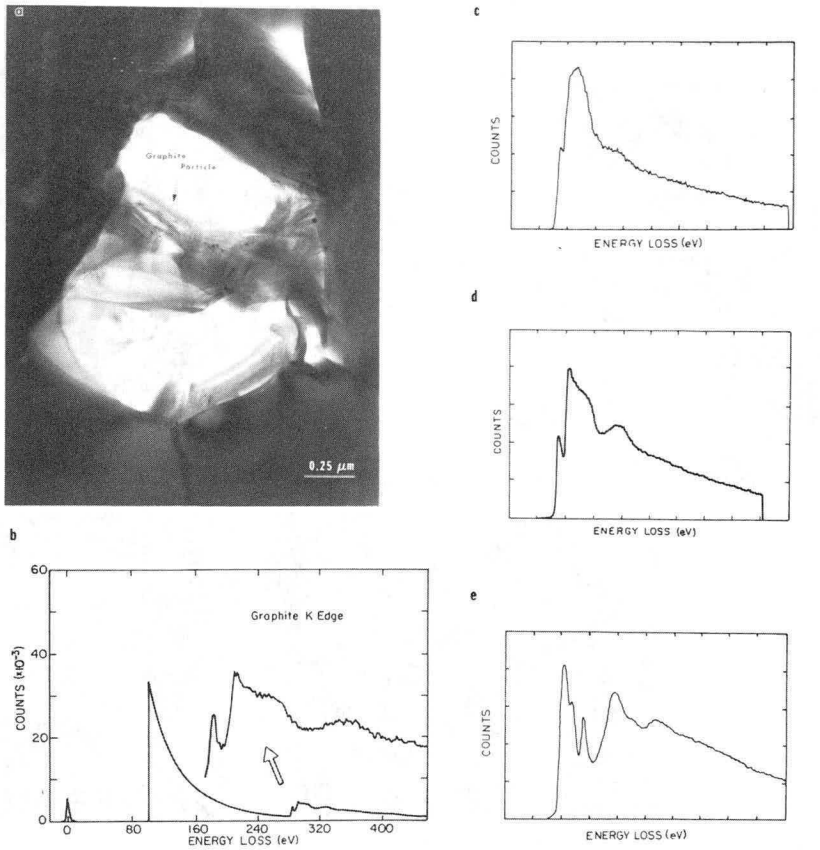
Fig. 13

Some Generic Microstructures: Ceramics

Grain Boundaries / Interfaces		Examples	Properties Limited
	Amorphous Films	Si_3N_4	Creep
	Partly Crystalline Films	Some Sialons	Creep
		Ferrites	Permeability
		Varistors	Voltage drop required
	Additives / Impurities	ß Na Alumina	Na^+ conduction
		$YBa_2Cu_3O_{7-x}$	Conduction a-b plane
		ZrO_2/Mullite Composites	Varied (creep, etc.)

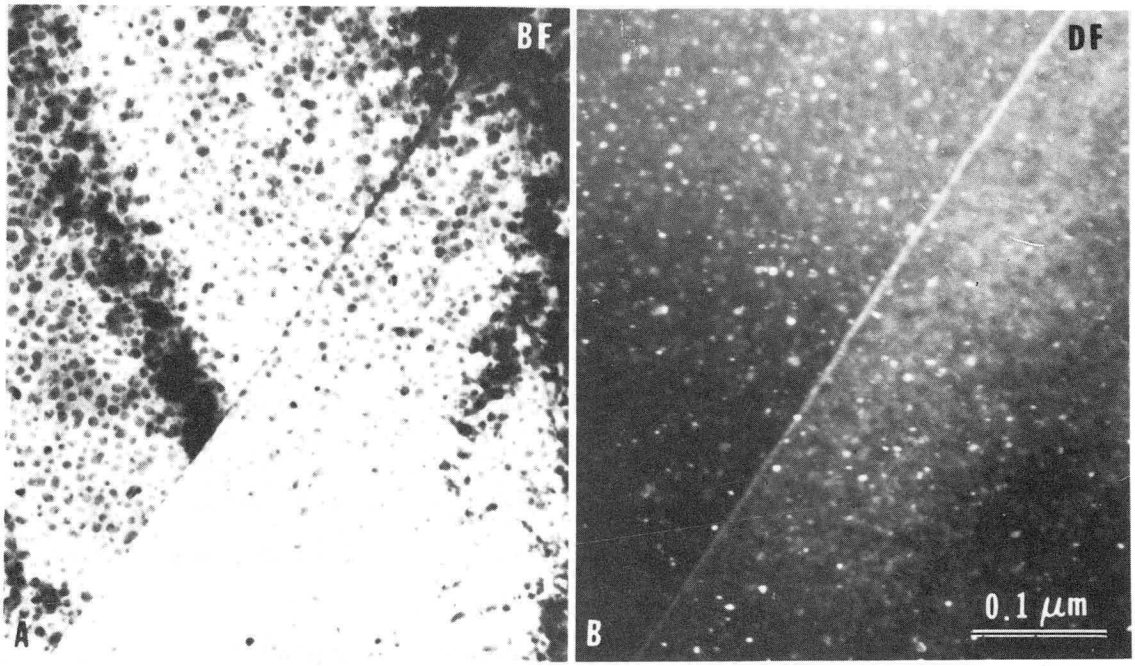
XBL 8712-5343

Fig. 14



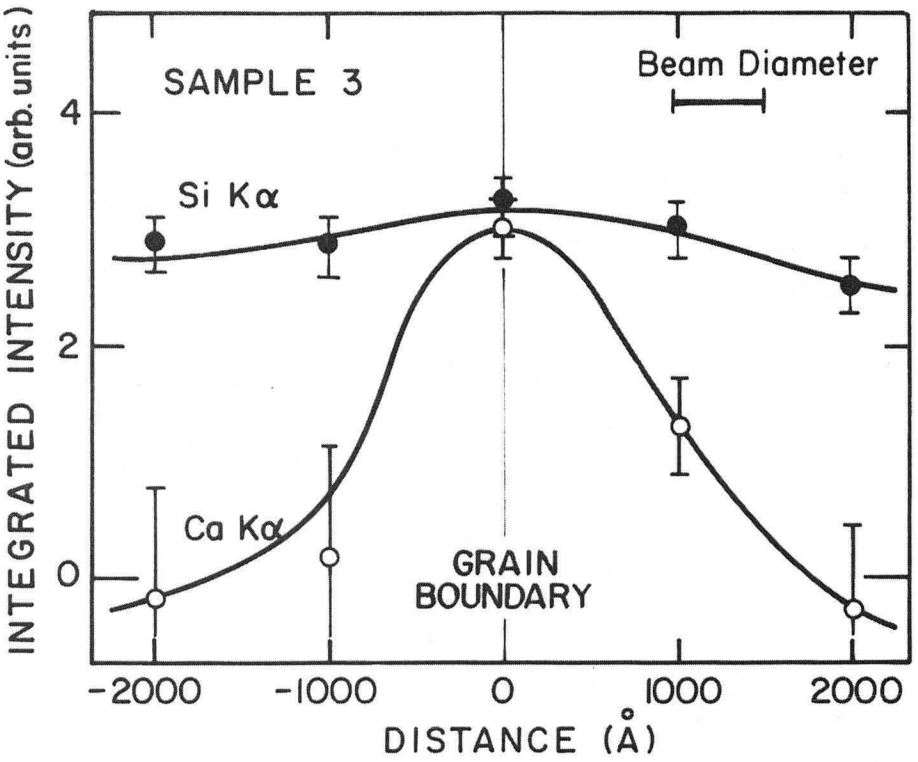
XBB 858-7008

Fig. 15



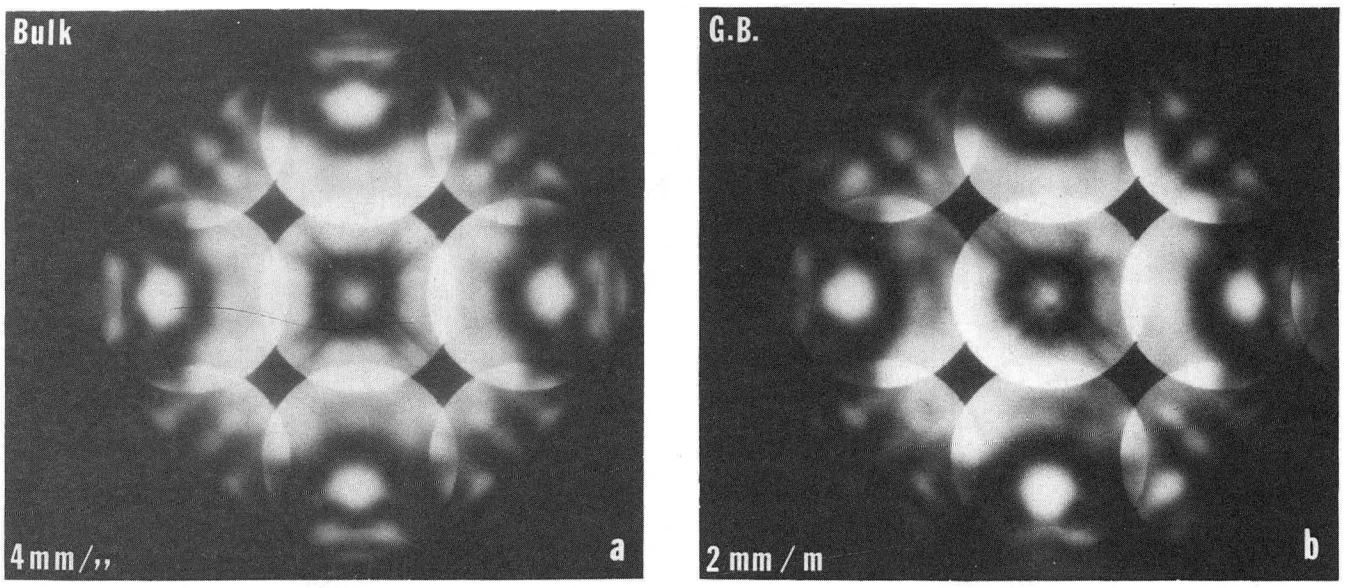
XBB 826-5216

Fig. 16a



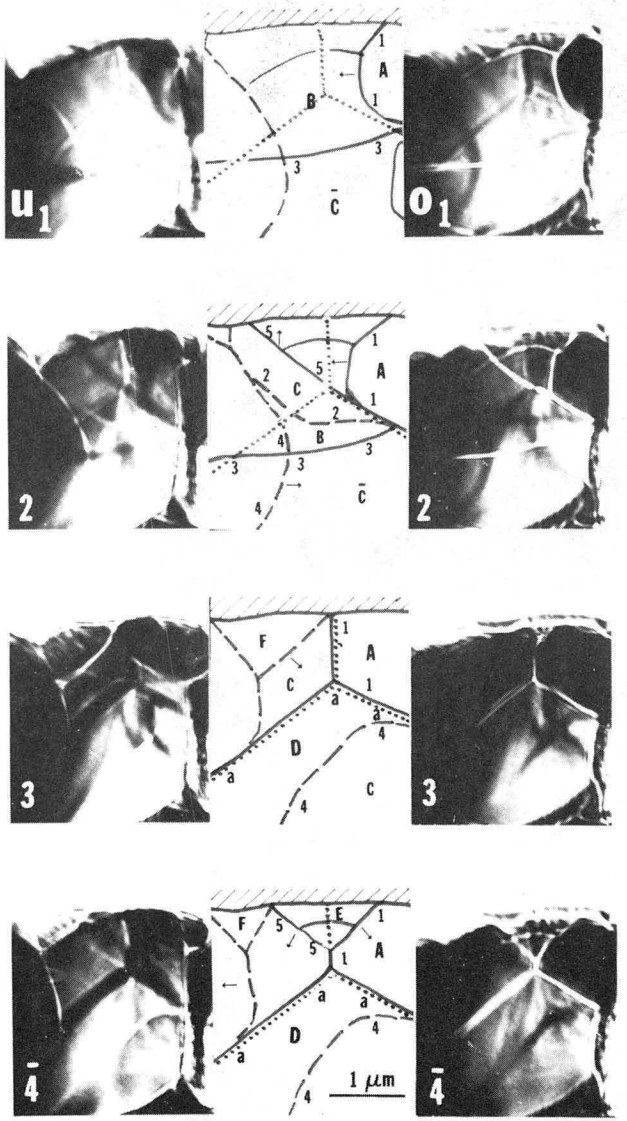
XBL 795-6375

Fig. 16b



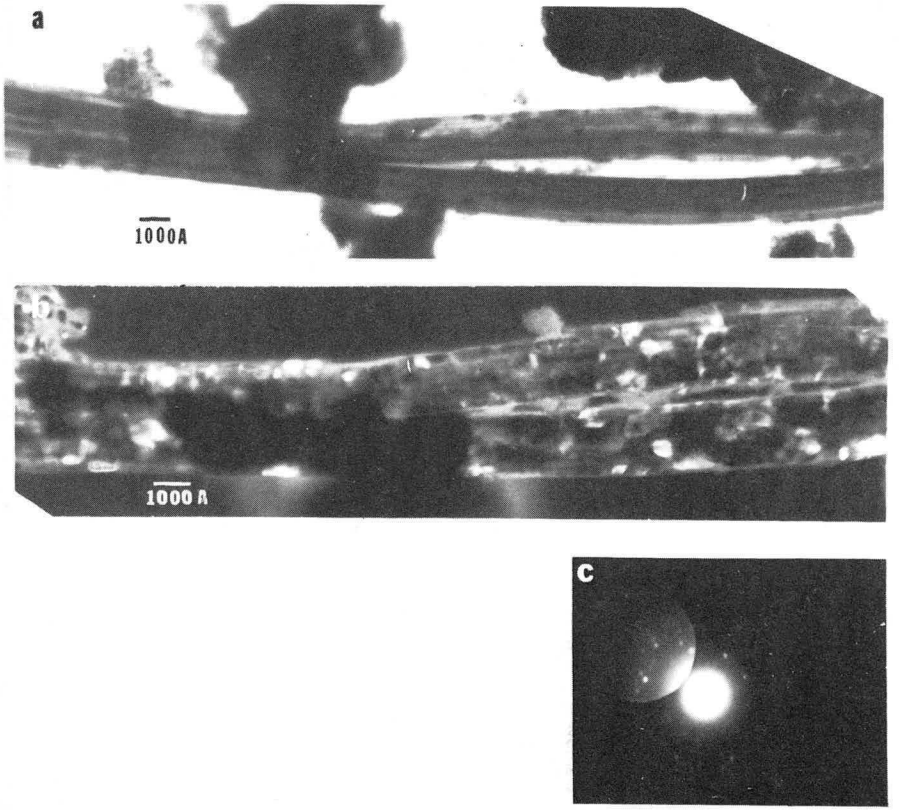
XBB 817-7040

Fig. 16c



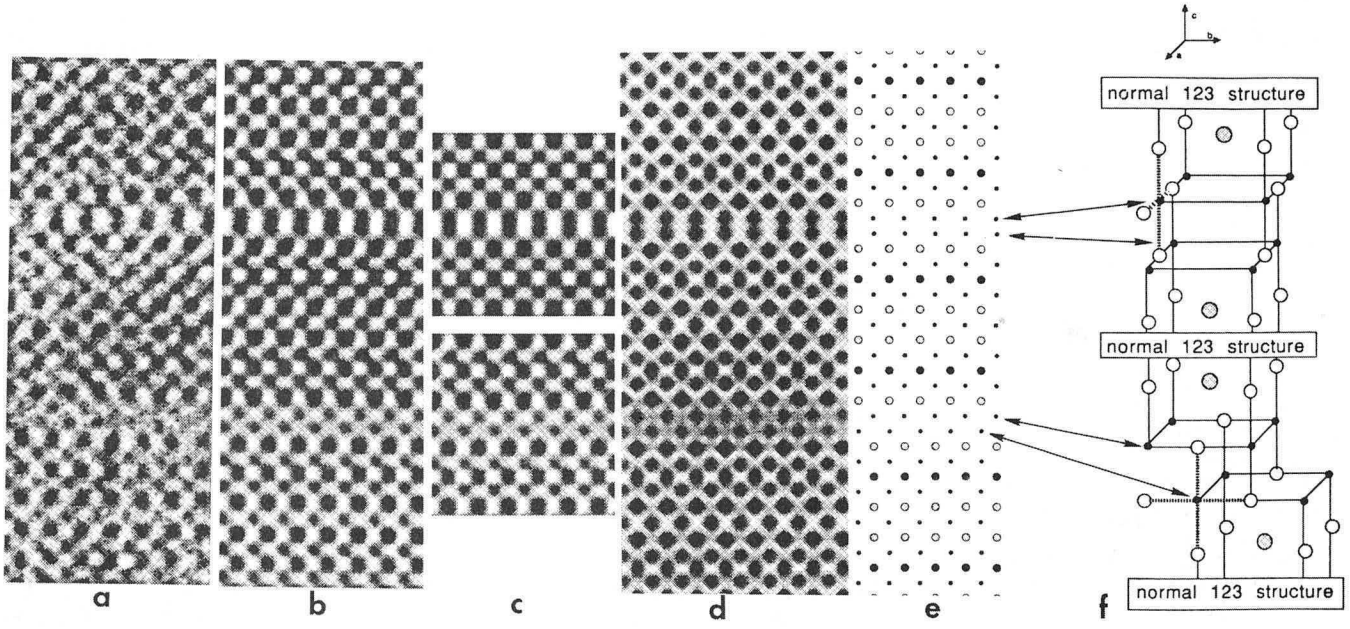
XBB 800-11966

Fig. 16d



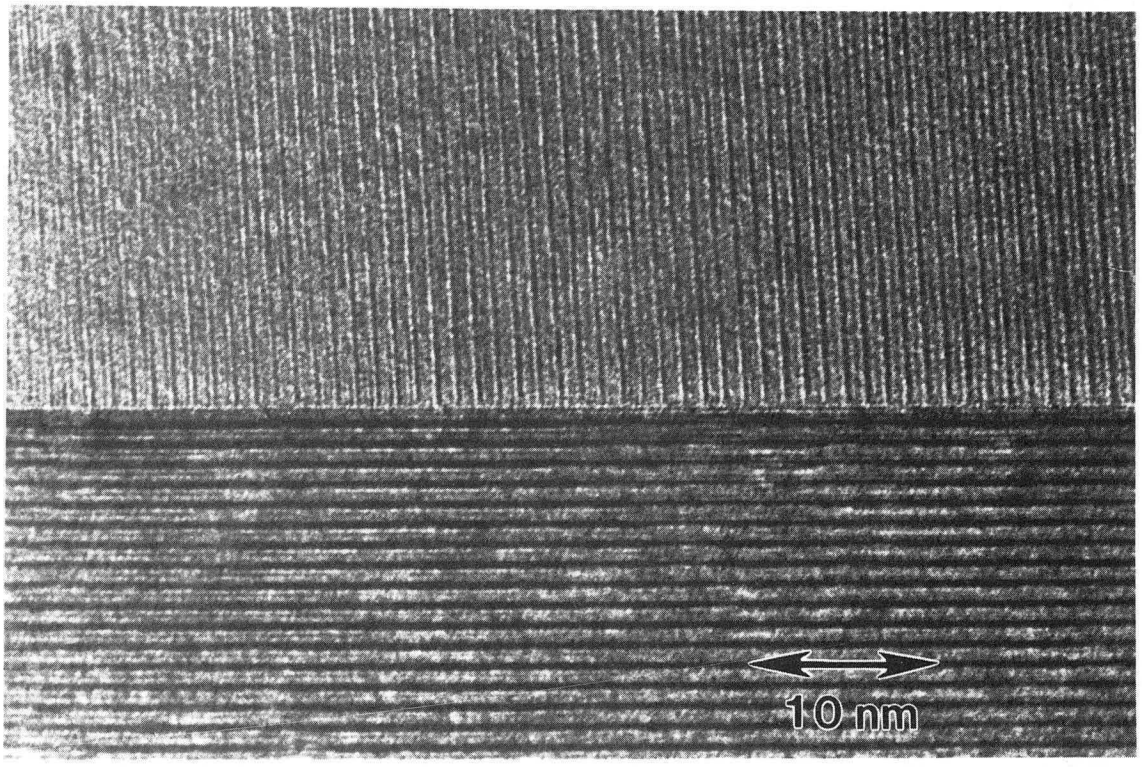
XBB 775-5414

Fig. 17



XBL 8711-4634

Fig. 18



XBB 870-10602

Fig. 19

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