In Situ Transmission Electron Microscope Studies of Irradiation-Induced and Irradiation-Enhanced Phase Changes*

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IN SITU TRANSMISSION ELECTRON MICROSCOPE STUDIES OF IRRADIATION-INDUCED AND IRRADIATION-ENHANCED PHASE CHANGES

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ABSTRACT

Motivated at least initially by materials needs for nuclear reactor development, extensive irradiation effects studies employing TEMs have been performed for several decades, involving irradiation-induced and irradiation-enhanced microstructural changes, including phase transformations such as precipitation, dissolution, crystallization, amorphization, and order-disorder phenomena. From the introduction of commercial high voltage electron microscopes (HVEM) in the mid-1960s, studies of electron irradiation effects have constituted a major aspect of HVEM application in materials science. For irradiation effects studies two additional developments have had particularly significant impact; (1) the availability of TEM specimen holders in which specimen temperature can be controlled in the range 10–2200 K and (2) the interfacing of ion accelerators which allows in situ TEM studies of irradiation effects and the ion beam modification of materials within this broad temperature range. This paper treats several aspects of in situ studies of electron and ion beam-induced and enhanced phase changes, including the current state of in situ ion beam capability internationally, and presents two case studies involving in situ experiments performed in an HVEM to illustrate the dynamics of such an approach in materials research.

INTRODUCTION

Transmission electron microscopes (TEMs) have become routine analytical tools in materials research over the past forty years or so. Analytical capabilities have evolved into an important array, allowing much more than the bright field (transmitted electron) imaging and selected area electron diffraction of the late 1940s and the 1950s, the most common including high resolution dark field (scattered electron) imaging, microarea diffraction (area defined by the electron probe rather than by an aperture in an image plan), convergent beam electron diffraction (CBED), X-ray energy dispersive spectroscopy (XEDS or just EDS) and electron energy loss spectroscopy (EELS). The good news is that each of these techniques has continued to be improved with respect to spatial resolution and sensitivity along with important improvements in the electronics, electron sources and the like, and consequently the range of applications of these instruments has continued to broaden; the bad news is that costs have seemed to skyrocket if one fails to consider the matter with reference to a constant currency (a basic TEM purchased in 1960 was in fact comparable in real cost to a TEM purchased today with the same functions and state-of-the-art electronics).

With the technical improvement of TEMs has come the development of a number of rather specialized instruments, especially in the past decade. It is convenient to now classify TEMs according to their primary function of (1) general analysis including imaging and diffraction and frequently elemental microanalysis, (2) high spatial resolution imaging—say 0.25 nm or better point-to-point, and (3) performing in situ experiments which are the instruments of emphasis in this presentation. This classification is illustrated in Figure 1. The boundaries separating the three categories of Figure 1 are rather fuzzy; for example, limited in situ experiments at high resolution are performed in several facilities, such as structure imaging during the thermal crystallization of Si, in which the structure dynamics of the crystal/glass
interface are of particular attention. A second and very important example is the category of TEMs dedicated to in situ experiments, during the conduct of which maximum analytical capability may be desired—structural, microstructural and elemental microanalysis.

In the first part of this presentation the essential requirements and strategies involved in in situ TEM studies will be reviewed with special emphasis on those studies involving ion irradiation and electron and ion irradiation. In the second part two particular studies will be described which illustrate these strategies.

In Situ Experiments

The term 'in situ' refers to experiments in which the relevant experimental techniques are employed while the experiment is being performed. An in situ experiment may be conducted truly dynamically, i.e., with the appropriate physical, chemical and structural analyses performed simultaneously as the phenomena occur, or, provided the phenomena under investigation allow, the experiment may be conducted incrementally, i.e., by interrupting the experiment to do the analyses. For example, an in situ experiment may be conducted incrementally if the time scale of relaxation phenomena, which counteract the phenomenon under investigation, is long compared to that of the targeted phenomenon itself.

Key elements in the execution of an in situ experiment in a TEM are (1) a microscope with intrinsically good resolution for the particular analyses required, optimally compromised to accommodate the experimental conditions, (2) specimen holder and stage systems exhibiting the highest degree of mechanical stability and experimental parameter control and measurement, (3) a specimen appropriately configured for the particular experiment and for the analytical techniques to be employed and, last but by no means least, (4) competent personnel. For example, in one experiment one might irradiate a specimen with high energy ions at 20 K while simultaneously measuring electrical resistance of the specimen and observing changes in the image or electron diffraction pattern. Provided the electrical resistance measurement and temperature control were automated, normally such an experiment would still require two people, one to perform the microscopy and the other to control the ion beam.
Table 1. Transmission electron microscopes with in situ ion-beam capability.

<table>
<thead>
<tr>
<th>Location and Investigators</th>
<th>Ion Accelerator</th>
<th>Interface</th>
<th>TEM</th>
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<tbody>
<tr>
<td>Argonne National Laboratory (Chicago) Allen Rahn</td>
<td>Acceleration: Tandem Voltage: 200-2000 kV</td>
<td>ES-deflector: 57 deg Final collimator: 2 mm</td>
<td>AEI EM 1200 100-1200 kV Ion Flux (max): $10^{13}$ cm$^{-2}$ s$^{-1}$</td>
</tr>
<tr>
<td>Kyushu University (Fukuoka) Kinoshita Yoshida</td>
<td>Acceleration: Ion implanter Voltage: 20-650 kV</td>
<td>ES-deflector: 57 deg Final collimator: 2 mm</td>
<td>KEssel EM 1200 1200 kV</td>
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<tr>
<td>Kyushu University (Kasuga) Yoshida Muroga</td>
<td>Voltage: &lt;1-10 kV Ion Source: Duoplasma</td>
<td>ES-deflector: 80 deg in EM</td>
<td>JEOL 1250 1250 kV</td>
</tr>
<tr>
<td>Hitachi Co. Ltd. Energy Research Inst. (Hitachi)</td>
<td>Voltage: 10-400 kV (Pulsed dual ion)</td>
<td>ES-deflector: 57 deg</td>
<td>Hitachi H-800 200 kV</td>
</tr>
<tr>
<td>Central Research Inst. of Electric Power (Kamak)</td>
<td>Voltage: 20 kV</td>
<td>Deflector: 0 deg</td>
<td>JEOL 200 CX 200 kV Ion Flux: $5\times10^{13}$ He cm$^{-2}$ s$^{-1}$</td>
</tr>
<tr>
<td>CSNSM-CNRS (Orsay) Rusult</td>
<td>Voltage: 5-200 kV</td>
<td>ES-deflector: 10 deg Final collimator: 5 mm</td>
<td>Phillips EM400 120 kV</td>
</tr>
<tr>
<td>Hokkaido University (Sapporo) Takahashi Omuki</td>
<td>Voltage: 20-300 kV</td>
<td>ES-deflector: 60 deg Final collimator: 3 mm</td>
<td>Hitachi H-1300 1300 kV Ion Flux (max): $10^{13}$ He cm$^{-2}$ s$^{-1}$</td>
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<tr>
<td>Japan Atomic Energy Research Institute (Tokai) Hojou</td>
<td>Voltage: 20 kV</td>
<td>ES-deflector: 72 deg Final collimator: 5 mm</td>
<td>JEOL 200C 200 kV</td>
</tr>
<tr>
<td>University of Tokyo (Tokai) Ishino Sekimura</td>
<td>Voltage: 10,20,30-400 kV</td>
<td>ES-deflector: 45 deg Final collimator: 4 mm</td>
<td>JEOL 200B 200 kV Ion Flux (max): $3\times10^{13}$ Ar cm$^{-2}$ s$^{-1}$</td>
</tr>
<tr>
<td>National Research Institute for Metals (Tsukuba) Furuya</td>
<td>Voltages: 100 kV; 30 kV (Simultaneous dual ion)</td>
<td></td>
<td>JEOL 200CFX 200 kV JEM ARM-1250 1250 kV (1993)</td>
</tr>
<tr>
<td>Institute of Physics and Chemistry (Wako) Yagi</td>
<td>Acceleration: Tandem Voltage: 1000-4000 kV</td>
<td>ES-deflector: 57 deg</td>
<td>Hitachi H-800 200 kV Ion Flux (max): $5\times10^{13}$ He cm$^{-2}$ s$^{-1}$</td>
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TEMs for In Situ Ion Beam Studies

One of the specialized types of TEMs which has been developed over the past two decades is that with in situ ion beam capability. Today more than four-fifths of the facilities employing TEMs with in situ ion beam capability are in Japan. Table 1 summarizes briefly the locations and a few essential aspects of the eleven installations in operation today in Japan (9), the USA (1) and France (1). These installations involve TEMs with usable electron accelerating voltages ranging from...
100 kV (Orsay) to 1.3 MV (Hokkaido University) and bombarding ion energies, from less than 1 keV (Kyushu University—Kasuga) to 16 MeV (Institute of Physics and Chemistry—Wako). One of the installations involves simultaneous dual ion capability (NRIM—Tsukuba) and another, alternating pulsed dual ion capability (Hitachi—Mito). One of the obvious advantages of having available in one place a broad range of ion energies, such as at Argonne, is the capability for performing ion-induced damage studies without having to employ self ions, as well as in situ ion implantation studies. Of these eleven installations, only three (Hokkaido, Kyushu and Argonne) utilize high voltage electron microscopes (HVEMs). The NRIM at Tsukuba will be replacing the present 200 kV TEM with a 1.25 MeV HVEM in 1993.

From the irradiation effects point of view, certainly the most important aspect of having a microscope with a broad range of electron energies is the corresponding ability, on the one hand, to create Frenkel pairs well above threshold for a broad spectrum of materials and, on the other hand, to perform, when necessary, the subsequent or intermittent analyses of the specimen at electron energies well below threshold. While electron accelerators had existed for several decades earlier, it was the introduction of HVEMs into materials science in the mid 1960s which made large electron flux and thus large electron dose studies possible, even with a W filament as electron source. Furthermore because the electron probe is also the imaging probe, one knows precisely the region of specimen being electron irradiated as well as the crystal orientation(s) of that region from electron diffraction.

As an example of the significance of the magnitude of electron flux in a TEM, the average electron current density corresponding to a 100 nA TEM probe of 2 μm diameter is 3.2 A cm⁻² or 2x10¹⁹ electrons cm⁻² s⁻¹. For a displacement cross-section of 50 barns for Frenkel pair production (e.g., 1 MeV electrons bombarding Ni), this electron flux produces an average damage production rate of 10⁻³ dpa s⁻¹ which experimentally is a very convenient order of magnitude.

Specimen Configuration

For in situ TEM experiments in which the analyses required are limited to imaging, diffraction and/or elemental microanalysis, usually no special specimen preparation is required beyond that for these analytical techniques. However, this is not always the case. For example, proper interpretation of structure images or of electron energy loss spectra may require that the specimen area be very thin (say, 10 nm or less), but this may be inconsistent with some other geometrical requirement of the experiment, such as sufficient specimen thickness in an ion or electron irradiation experiment so that the free surface is not everywhere the dominant sink for point defects.

In some in situ experiments, specialized specimen geometries may be required, either because of some particular analytical technique to be employed or because of the experiment itself. An example of the first is the thin film specimen employed in conjunction with electrical resistance measurement during electron irradiation [1]. In such a case the gauge section must be small enough for a reasonably large and uniform electron flux during the experiment, and the specimen must have appropriate electrical lead pads.

In some cases, the specimen configuration is dictated by the experiment itself. Usually when in situ ion irradiation experiments in a TEM are performed, the specimen and hence the resulting phenomena are seen essentially in plan view. The irradiation and viewing directions are more or less normal to the surface of the specimen. In addition, because of the requirement that the specimen be relatively thin, a few tenths of μm or less, which is a distinct advantage when high energy implantation effects are to be avoided, such deep implantation type experiments have not been possible. Geometrically at least, these problems can be circumvented by ion irradiating the specimen at 90 degrees with respect to the
viewing direction as illustrated in Figure 2; i.e., by irradiating an edge of the specimen, parallel to, rather than normal to, the major surfaces of the specimen. This turns the specimen into a kind of dynamic cross-section specimen as far as the observer is concerned. While there are a number of problems with actually doing this and also with interpreting the results quantitatively, the results of such an experiment, shown briefly in Figure 3, demonstrate the principle [2]. The experiment involves the amorphization of Si at room temperature during edge bombardment (left side) by 1.5 MeV Kr ions. The HVEM was operated at 150 kV to avoid displacements due to electrons. The specimen is approximately 300 nm thick. The variation of damage with dose and with distance from the irradiated surface is evident.

Figure 3. Amorphization of a Si thin film, approximately 300 nm thick, during edge-on irradiation by 1.5 MeV Kr to (a) 1x10\(^{18}\) m\(^{-2}\), (b) 1.5x10\(^{18}\) m\(^{-2}\) and (c) 2x10\(^{18}\) m\(^{-2}\).

**Ion and Electron Dosimetry**

A critically important aspect of quantitation in irradiation experiments is the determination of particle fluxes and total doses. Typical architecture of a TEM equipped with Faraday cups for both ion and electron dosimetry is shown schematically in Figure 4. The electron dosimetry may be based on two Faraday cups situated within the microscope column which serve quite different functions. The upper Faraday cup (No. 1) in Figure 4 is located between the condenser lens system and the objective lens and is employed to determine total electron current incident on the specimen. A Faraday cup in this position is used mainly in conjunction with fundamental yield studies for electron energy loss spectroscopy
Figure 4. Schematic of ion and electron dosimetry systems in a TEM column with in situ ion beam capability.

(EELS) and the like. Essential to quantitative electron irradiation effects studies is the lower Faraday cup (No. 2) in Figure 4, located most conveniently in the viewing chamber below the final projector lens. This Faraday cup is employed to profile the electron beam intensity $I_e(x,y)$ to be employed in an electron irradiation. This cup is moveable in the x-y plane and employs typically a 2 mm aperture to define that portion of the electron beam entering the cup. Such measurements are made with the objective aperture withdrawn and with no part of the specimen in the electron beam. During an electron irradiation experiment the screen current is usually employed to monitor not only the position of the electron beam with respect to some fixed feature of the specimen but also to monitor the constancy of the beam current striking the screen during the experiment. This practice must be employed with great caution whenever changes in structure, thickness, orientation or contamination of the specimen area imaged may occur during the irradiation, which change the image intensity without any change in electron intensity incident on the specimen.

Also shown in Figure 4 is the ion dosimetry system, which consists of two parts, a fixed ion skim cup, employed in maintaining the constancy of ion beam position and intensity and in determining the ion dose as a function of time during an ion irradiation experiment, and a removeable Faraday cup employed to establish the ion flux immediately prior to and after the ion irradiation of the specimen. To mitigate effects of ion beam divergence from the defining aperture of the ion dosimetry system, the beam defining aperture is situated as close to the specimen as possible and at the same position along the ion beam path as the defining aperture of the ion Faraday cup. Usually this can be within a few cm of the specimen.

IN SITU ION IRRADIATION AND STUDIES OF PHASE TRANSFORMATIONS

Rather extensive and important studies of effects of electron and ion irradiation on solid state phase transformations in materials have been conducted which have employed in situ TEM techniques. These include, for example, electron and ion irradiation-induced amorphization of intermetallics [e.g., 3, 4, 5, 6], radiation-
induced segregation/precipitation [e.g., 7], and irradiation-induced crystallization [e.g., 8, 9, 10].

The importance of in situ TEM in such studies is threefold; (1) the experiments can be performed rather quickly, often with a stream of simultaneous analytical results; (2) particularly for experiments above or below room temperature, analysis at temperature may be essential to avoid possible mitigating phenomena during warmup or cooldown; and (3) realtime observation of one region may be essential to understanding the mechanism or mechanisms of a particular phenomenon. In the second part of this presentation, examples of several particular experiments are cited which illustrate these three points. The experiments were performed at Argonne's HVEM-Tandem Facility, the major equipment of which appears in Table 1 as the first entry [11].

Ion Irradiation-Induced Amorphization and Enhanced Crystallization in Cr+30 at pct Ti [8, 12]

In an in situ experiment, the specimen holder is a key element of a microlaboratory within which, ideally, an entire experiment can be performed, including all required aspects of analysis. In this first example, a single tilt, helium cooled specimen holder is employed for a low temperature ion irradiation experiment involving a Cr+30 at pct Ti alloy.

Following equilibration at 1500 K and quenching to room temperature, this alloy consists of a Laves phase (ordered TiCr2) and the Cr-rich bcc phase \( \beta \) (~23 at pct Ti). 1 MeV Kr irradiation at temperatures < 250 K amorphizes the intermetallic but not the \( \beta \) phase; with prolonged irradiation, the amorphous material (a-TiCr2) crystallizes to \( \beta \) (denoted \( \beta_r \), ~33 at pct Ti). Ion irradiation at room temperature also causes the \( \beta \) phase to grow by migration of the bcc/intermetallic interface to produce \( \beta_r \). Figure 5 shows lattice fringe images of Laves phase before ion irradiation and during 1 MeV Kr irradiation at a dose of about 6x10\(^{17}\) m\(^{-2}\). The original structure is still more or less recognizable during ion irradiation but the electron diffraction pattern reveals a diffuse ring, suggesting that the disorganization which is evident in Figure 5b is associated with amorphous zones in the Laves phase, formed above the amorphization temperature limit prior to conversion of the Laves phase to the bcc structure. The spacing of most of the fringes in Figure 5a is 2.4 nm, the periodicity of this Laves phase in [001].

The purpose of the low temperature experiment with this alloy was to determine the effect of ion irradiation temperature on the rate of conversion of a-TiCr2\( \rightarrow \beta_r \). The strategy in this case is straightforward. A normal TEM thinned foil is employed,
Figure 6. 1 MeV Kr irradiation-induced crystallization of amorphized TiCr$_2$: (a) 100 K, total dose 1.2x10$^{19}$ m$^{-2}$; (b) 130 K, total dose 1.4x10$^{19}$ m$^{-2}$ and (c) 145 K, total dose 1.8x10$^{19}$ m$^{-2}$. (d) Specimen warmed to room temperature from 145 K without further ion irradiation.

which is clamped on an In washer in the helium holder to ensure good thermal contact. A single specimen is sufficient for the entire experiment and the evolution of microstructure in a single area in which both α-TiCr$_2$ and β are visible is to be followed. The HVEM is operated at 500 kV. The specimen is cooled to 100 K and irradiated with 1 MeV Kr at a flux of 8x10$^{15}$ m$^{-2}$s$^{-1}$. The Laves phase is completely amorphized by a dose of 10$^{18}$ m$^{-2}$. The image is recorded periodically during the irradiation. The microstructure after 1.2x10$^{19}$ m$^{-2}$ is shown in Figure 6a. The temperature is changed and the irradiation resumed. In this particular experiment the irradiation temperatures were 100, 130 and 145 K as indicated by the Pt resistance thermometer on the specimen holder.

Subsequently the α-TiCr$_2$/β interface velocity is determined from the micrographs. For example, at 145 K the interface velocity is 9x10$^{-11}$ m s$^{-1}$ which corresponds to an effective damage zone created by each Kr ion approximately 3 nm in diameter. In the range of temperature 80–180 K the interface velocity is found to follow Arrhenius behavior with irradiation temperature with an activation energy of 0.027 eV. Thus from the micrographs of a single day’s experiment it was possible to determine the ion irradiation-induced growth rate of the β for a constant ion flux as a function of temperature, to establish an effective activation energy for the growth process and to deduce an effective volume transformed per ion. The entire experiment involved one area of the specimen for observation. Such direct fine scale observation of the time-resolved evolution of a particular region of a microstructure is the unique characteristic of in situ TEM experiments.
One final point is illustrated by Figure 6d which shows the microstructure of the region observed during the low temperature experiment, after the specimen has returned to room temperature and the remaining a-TiCr2, crystallized. One would be hard pressed to deduce what had really happened during such an experiment if only initial and final microstructures observed at room temperature were available.

**Ion and Electron Irradiation Enhanced Crystallization in Non-Crystalline CoSi2 Thin Films [10, 13, 14]**

In this second example, the effects of ion irradiation, electron irradiation and thermal annealing are employed in a study of the crystallization of coevaporated (non-crystalline) thin films of composition CoSi2. Because the effects of particle irradiation in the processes of nucleation and growth are fairly complex in this material, just three particular aspects of the study are chosen here to illustrate the conduct of such experiments in the HVEM environment. The specimens were coevaporated thin films ~40 nm thick, covering ~2.3x10^-6 m^2 windows in thick Si substrates. Observation of the specimen in the HVEM during ion irradiations and during thermal growth experiments was conducted with the HVEM operating at 120–150 kV, well below threshold for displacement by electrons.

The phenomenon of ion-assisted crystal growth is illustrated in Figure 7. The specimen had been thermally crystallized partially prior to ion irradiation with 1 MeV Kr. Figure 7a shows the microstructure after 3.4x10^{18} m^{-2} and Figure 7b, after 6.8x10^{18} m^{-2}, the irradiation being performed at 300 K. The existing large crystals have grown and a profusion of small crystals has appeared. This was the first ion irradiation experiment we performed with this material. Within a two hour experiment it was possible not only to establish that further experiments of this kind should be performed but also to determine that on average the increase in volume associated with this ion-assisted growth at 300 K is approximately 4x10^{-26} m^3 per ion (40 nm film). Subsequent experiments revealed that the profusion of crystals in Figure 7b was not primarily the result of ion irradiation induced nucleation in this specimen but rather ion irradiation induced growth of nuclei which had been produced during observation with a 300 kV TEM of the prior thermal growth process in this particular specimen [3]. Furthermore, even in the absence of prior electron irradiation above threshold, it is necessary to thoroughly ion beam mix the as-deposited silicide at a low temperature in order to eliminate crystal embryos, if one wishes to examine irradiation-induced nucleation of crystallization.

**Figure 7.** Ion irradiation assisted crystallization of CoSi2 thin film at 300 K (fine dispersion of crystals), following partial thermal crystallization in a 300 kV TEM (massive crystals). 1.5 MeV Kr irradiation to doses of (a) 3.4x10^{18} m^{-2} and (b) 6.8x10^{18} m^{-2}. 
An example of a portion of such an experiment is shown in Figure 8. The left half of the specimen area shown in Figure 8 had been irradiated to a dose of $3 \times 10^{19}$ m$^{-2}$ at 90 K, which has caused obvious thinning of the film by sputtering. The film was then thermally crystallized at 450 K without additional ion irradiation. Figure 8a shows the area at 25 minutes of annealing; Figure 8b, at 80 minutes. "Normal" thermal crystallization occurs in the non-irradiated portion of the film, evidently from embryos in the as-deposited material. No nucleation was observed to occur in the ion irradiated area. However, crystals grew from the non-irradiated area into the irradiated area with undiminished growth rate (about 0.6 nm s$^{-1}$). In addition there is a remarkable change in strukturale of the crystals as they grow into the prior irradiated region, from a large, equiaxed structure to a narrow, columnar structure. The range of misorientations in a given crystal, however, is very similar in the two regions. This experiment involved two specimen holders, the first, a low temperature holder with which the 90 K ion irradiation was performed, and the second, a heating holder in which the annealing process was performed. In this case the outcome of the experiment is unaffected by this, provided of course the specimen is not destroyed in the process of being transferred from one holder to another. The entire experiment consumed approximately five hours. The results of a similar experiment in which the ion beam mixing was accomplished with 300 kV Si rather than 1.5 MeV Kr was somewhat different, at least partly because the implantation of additional Si complicates the crystallization process. Both Si and silicide are formed, which alters both the silicide morphology and the overall kinetics of crystallization.

In the previous experiment, ion irradiation was performed at 90 K, prior to thermal annealing, in order to eliminate crystal embryos in the as-deposited specimen. The results of a similar experiment in which the ion irradiation is performed at 300 K, prior to thermal annealing, are shown in Figure 9. The lower third of the specimen area shown was irradiated with 1.5 MeV Kr at a flux of approximately $7 \times 10^{15}$ m$^{-2}$s$^{-1}$ to a dose of $3.4 \times 10^{18}$ m$^{-2}$ at 300 K. The specimen was then heated to 450 K for thermal crystallization without further ion irradiation. The micrographs shown in Figure 9 were recorded at 2 minute intervals. The number of crystals per unit area is approximately the same in the irradiated and non-irradiated regions, but those in the ion irradiated region grow about three times faster, suggesting that defects introduced in the "amorphous" starting material by ion irradiation enhance the short range rearrangements necessary for crystal growth. This experiment required about two hours.
Figure 9. Thermal crystallization sequence at 2 minute intervals for a CoSi$_2$ thin film at 450 K. Lower third of region had been subjected to 1.5 MeV Kr irradiation to $3.4 \times 10^{18}$ m$^{-2}$ at 300 K prior to thermal anneal.

CONCLUDING REMARKS

In situ experiments in TEMs are employed in two ways: first, to obtain detailed information from a region of specimen as its microstructure and physical and chemical properties evolve during some specific treatment and, second, to quickly survey the effects of varying one or more parameters in an experiment in order to determine a range of the parameter(s) within which a phenomenon is to be investigated, perhaps by some complementary experimental techniques. While the examples in this presentation have been drawn exclusively from irradiation effects in conjunction with two types of phase transformations, amorphization and solid state crystallization, they are just typical examples in these areas. Furthermore the area of effects of electron and ion irradiation on phase transformations is an enormously broad one, even considering only that part which has relied on the specific techniques for dynamic observation described here. The major focus of this presentation has been to present an overview of dynamic TEM experiments from which relevant experimental data flow and are collected essentially simultaneously during the experiment and from a particular region of continuous observation. Several examples have been presented to illustrate the strategies involved in the conduct of such an experiment in the area of electron and ion irradiation-effected phase changes, with emphasis on amorphization and solid state crystallization.

ACKNOWLEDGMENT

The author acknowledges the support of the HVEM Tandem Facility at Argonne National Laboratory, which is a user facility funded by the U. S. Department of Energy, BES—Materials Sciences, under Contract W-31-109-Eng-38. The Facility is available to the scientific community, usually free of charge for non-proprietary research [11].

REFERENCES

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