Real time observation of reconstruction transition on GaAs (111)B vicinal surface by scanning electron microscopy

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Abstract

Scanning electron microscopy (SEM) has been applied to observe directly the $\sqrt{19}\times\sqrt{19}$ and $(1\times1)_{HT}$ reconstructions and the transition associated step bunching on the GaAs (111)B surfaces under As pressure. Close to the transition point, $\sqrt{19}\times\sqrt{19}$ and $(1\times1)_{HT}$ reconstructions are observed in dark and bright domains by SEM and determined by micro-probe reflection high-energy electron diffraction (μ-RHEED). The reconstruction transition diagram shows hysteresis. The stepped surface morphology during the reconstruction transition was unstable. Heavy step bunching with rough macrostep edges was observed.

Real time observations of reconstruction transitions on III-V semiconductor surfaces have been done mainly by RHEED. Recently the transitions between 2X4 and 4X2 reconstructions on InAs (001) surface have been directly observed by SEM which is equipped in a molecular beam epitaxy (MBE) system [1]. In this paper we report the real time imaging of the domain formation during the reconstruction transitions between $\sqrt{19}\times\sqrt{19}$ [2,3] and $(1\times1)_{HT}$ [2] on the GaAs (111)B vicinal surfaces obtained in a μ-RHEED/SEM MBE system. Step bunching associated with the reconstruction transition was studied.

The μ-RHEED/SEM MBE system used for the present study has been described elsewhere [4]. During the observation, a 25 keV electron beam was incident by a glancing angle of 10^{o} onto the sample surface with the azimuth along the $[\bar{1}10]$ direction, and the secondary electron image of the surface with dynamic focus and slight tilt compensation was obtained on the screen in real time. GaAs (111)B substrates inclined $1^{o}\pm0.1^{o}$ toward the $[\bar{1}\bar{1}2]$ direction were employed for the study. After a GaAs buffer layer was grown, a surface covered by macrosteps was obtained and the macrostep edges were intended to be used to visualize the contrast on the surface. Then the surface reconstructions and the transitions were observed by varying the temperature with a changing rate of 3 to 5 ^{o}C/min under a constant As pressure of about 2×10^{-6} Torr.

In the first experiment, a macrostepped surface was obtained by the growth at 630 ^{o}C with a growth rate of 5 nm/min. The surface reconstruction after the termination of the growth as well as during the growth was found being $(1\times1)_{HT}$ by μ-RHEED. Then the temperature was lowered down slowly and kept constant at 624 ^{o}C. It was found that dark domains were formed immediately to certain sizes as was shown in **Fig. 1**. The steps proceed down from the left to the right. Except some noises in the figure, the dark domains always started to appear from the macrostep edges and developed on to the lower terraces. It was found that the domains were mostly confined when they met the next macrostep edges although a few of them could overcome the step barriers and develop onto the next terraces. By employing μ-RHEED which could focus the electron beam down to a few nanometers, it was confirmed that the dark domains were of Ga-dorminated $\sqrt{19}\times\sqrt{19}$ reconstruction, and those areas with bright

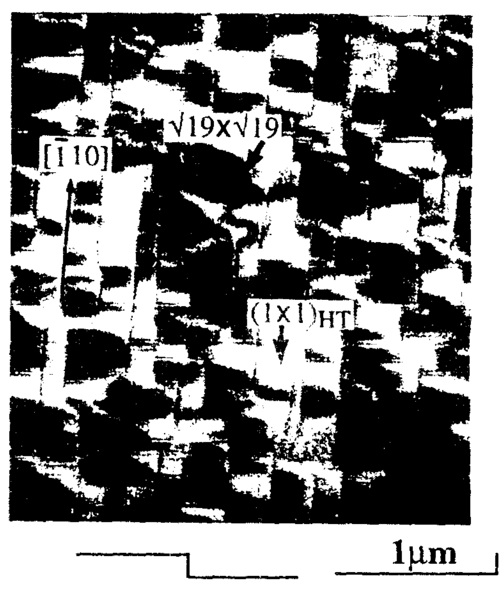

Fig. 1. Macrostepped GaAs (111)B vicinal surface during the growth interruption at 624 ^{o}C. $\sqrt{19}\times\sqrt{19}$ reconstructed dark domains were developed from the macrostep edges on to the $(1\times1)_{HT}$ reconstructed lower terraces from the left to the right.

contrast were of Ga-rich $(1\times1)_{HT}$ reconstruction.

In the second experiment, transitions of surface reconstructions between $\sqrt{19}\times\sqrt{19}$ and $(1\times1)_{HT}$ were observed in real time by changing the annealing temperature around the transition point of 625 °C. Since it takes 80 second for the electron beam to finish the scanning of one SEM image, the vertical axis gave a temperature dependence during acquiring information on the surface reconstruction transitions. An as-grown surface with small and wavy macrosteps under $\sqrt{19}\times\sqrt{19}$ reconstruction at 620 °C is shown in **Fig. 2a**. The step down direction is from the left to the right and the average macrostep spacing was about 200 nm. At first, the temperature was raised up gradually. While passing 625 °C, it was found that the secondary electron intensity was suddenly increased. The surface was split into bright and dark domains confined by the terrace sizes, as shown in **Fig. 2b** and **Fig. 2c**. With the increase of temperature, the number of dark domains decreased slowly. Finally they were completely removed when the temperature was increased to 635 °C, leaving a surface with rough step edges and islands as shown in **Fig. 2d**. After this transition, the average macrostep spacing was increased remarkably to about 350 nm. It is obvious that the step edges are of poor contrast in the bright background as is compared with that of **Fig. 2a**.

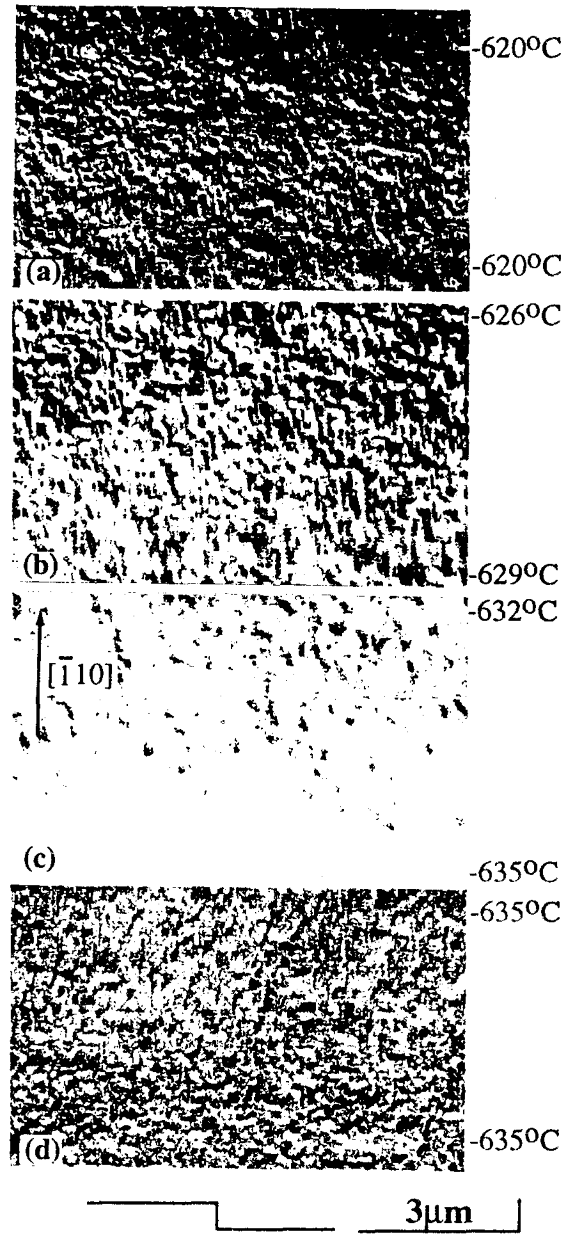

Fig.2. SEM images of phase transition from $\sqrt{19}\times\sqrt{19}$ to $(1\times1)_{HT}$ reconstruction on GaAs (111)B vicinal surface by raising the temperature during the growth interruption. **(a)**. a $\sqrt{19}\times\sqrt{19}$ reconstructed surface at 620 °C. **(b)** and **(c)**. $\sqrt{19}\times\sqrt{19}$ reconstructed dark domains were slowly eliminated above the transition point. **(d)**. a bright $(1\times1)_{HT}$ reconstructed surface was obtained at 635 °C. Steps proceed downward from left to right, and it takes 80 seconds to acquire a photo.

During the short annealing at 635 °C, islands on the large terraces were gradually removed and step edges became less wavy. The macrostep sizes increased to be about 400 nm as shown in the upper part of **Fig. 3a**. Then, the temperature was lowered down to pass through the transition point again. When the temperature was lowered to 625 °C, the transition suddenly occurred with the dark domains quickly developed as shown in the lower part of **Fig. 3a**. The remaining small potion of bright domains adjacent to the macrosteps on upper terraces were finally eliminated until the temperature was lowered down to 620 °C, leaving a surface as shown in **Fig. 3b**. The macrostep edges changed back to bright contrast in the dark background. This transition process occurred quickly and no appreciable increase in the average macrostep spacing was observed.

Based on the above observation, a phase-diagram in the surface coverage of domains around the transition point was obtained which showed hysteresis. After further transition processes, the terrace sizes increased slowly, some small size islands appeared on the large terraces, and macrostep edges became even rougher.

For compound semiconductors, it is difficult to analyze how the surface phase transition induces mass transfer because two kinds of atoms should be considered for the surface diffusion. When lowering the temperature from $(1\times1)_{HT}$ reconstructed surface down to $\sqrt{19}\times\sqrt{19}$ reconstruction, the

$\sqrt{19}\times\sqrt{19}$ domains develop quickly from the macrostep edges on to the lower macro-terraces along the step-down direction. Perhaps arsenic incorporation plays a major role to break the $(1\times1)_{HT}$ into $\sqrt{19}\times\sqrt{19}$ domains. On the contrary, when raising the temperature to pass through the transition point, the process is slow and it needs a stronger driving force to eliminate the $\sqrt{19}\times\sqrt{19}$ domains. Probably the diffusion of surface Ga atoms toward the step-down direction plays the major role except for arsenic evaporation. This resulted in the development of Ga-rich $(1\times1)_{HT}$ domains starting from the macrostep edges on to the upper terraces and obvious increase of macrostep sizes. A detailed analysis of mass transfer related to the reconstruction transitions is under further investigation.

In conclusion, Ga-stabilized $\sqrt{19}\times\sqrt{19}$ and Ga-rich $(1\times1)_{HT}$ reconstructions on the vicinal GaAs (111)B surface and the transitions between the two reconstructions have been observed directly by SEM under As pressure. The $\sqrt{19}\times\sqrt{19}$ domains are formed quickly from the macrostep edges on to the lower $(1\times1)_{HT}$ reconstructed terraces when the temperature is cooled down to pass through the transition point, and they are removed slowly from the step edge areas by raising the temperature. The resulted phase-diagram in the surface coverage of domains around the transition point shows hysteresis. Macrostep sizes are increased remarkably after the reconstruction transitions.

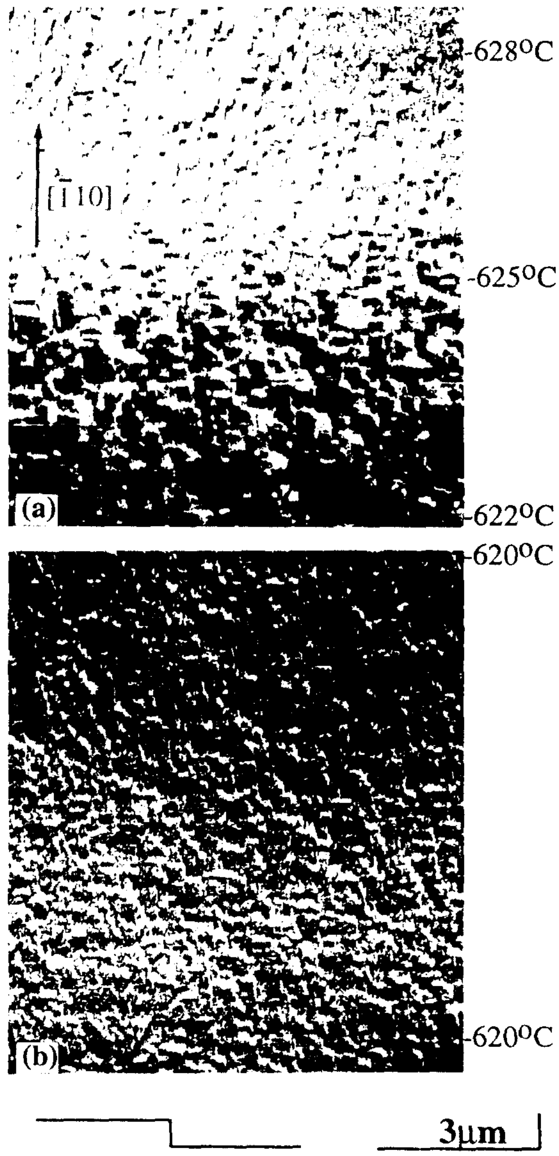

Fig.3. SEM images of the phase transition from $(1\times1)_{HT}$ back to $\sqrt{19}\times\sqrt{19}$ reconstruction on GaAs (111)B surface by lowering the temperature from 635 °C. **(a)**. when the temperature was passing the transition point, dark $\sqrt{19}\times\sqrt{19}$ domains were developing quickly. **(b)**. a $\sqrt{19}\times\sqrt{19}$ reconstructed surface was obtained at 620 °C.

References

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3. D.K. Biegelsen, R.D. Bringans, J.E. Northrup, and L.-E. Swartz, Phys. Rev. Lett. 65 (1990), 452.
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REAL TIME OBSERVATION OF RECONSTRUCTION TRANSITION ON GaAs (111)B VICINAL SURFACE BY SCANNING ELECTRON MICROSCOPY

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1. Introduction

2. Experiments

3. Discussion

4. Conclusions

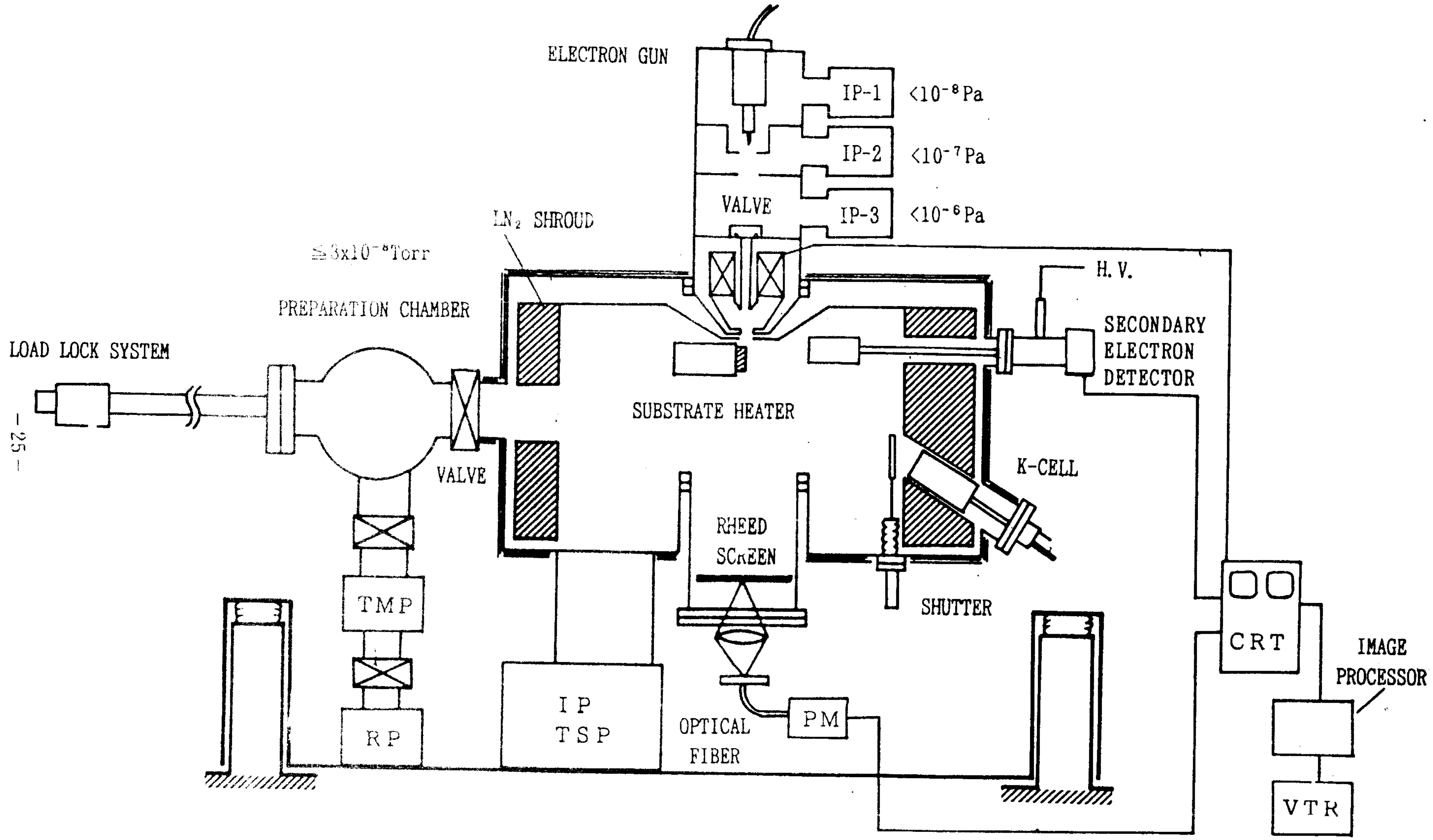
ELECTRON GUN
IP-1
<10⁻⁸ Pa
IP-2
<10⁻⁷ Pa
VALVE
IP-3
<10⁻⁶ Pa
LN₂ SHROUD
≦3x10⁻⁸Torr
PREPARATION CHAMBER
LOAD LOCK SYSTEM
H. V.
SECONDARY
ELECTRON
DETECTOR
SUBSTRATE HEATER
VALVE
K-CELL
RHEED
SCREEN
SHUTTER
TMP
RP
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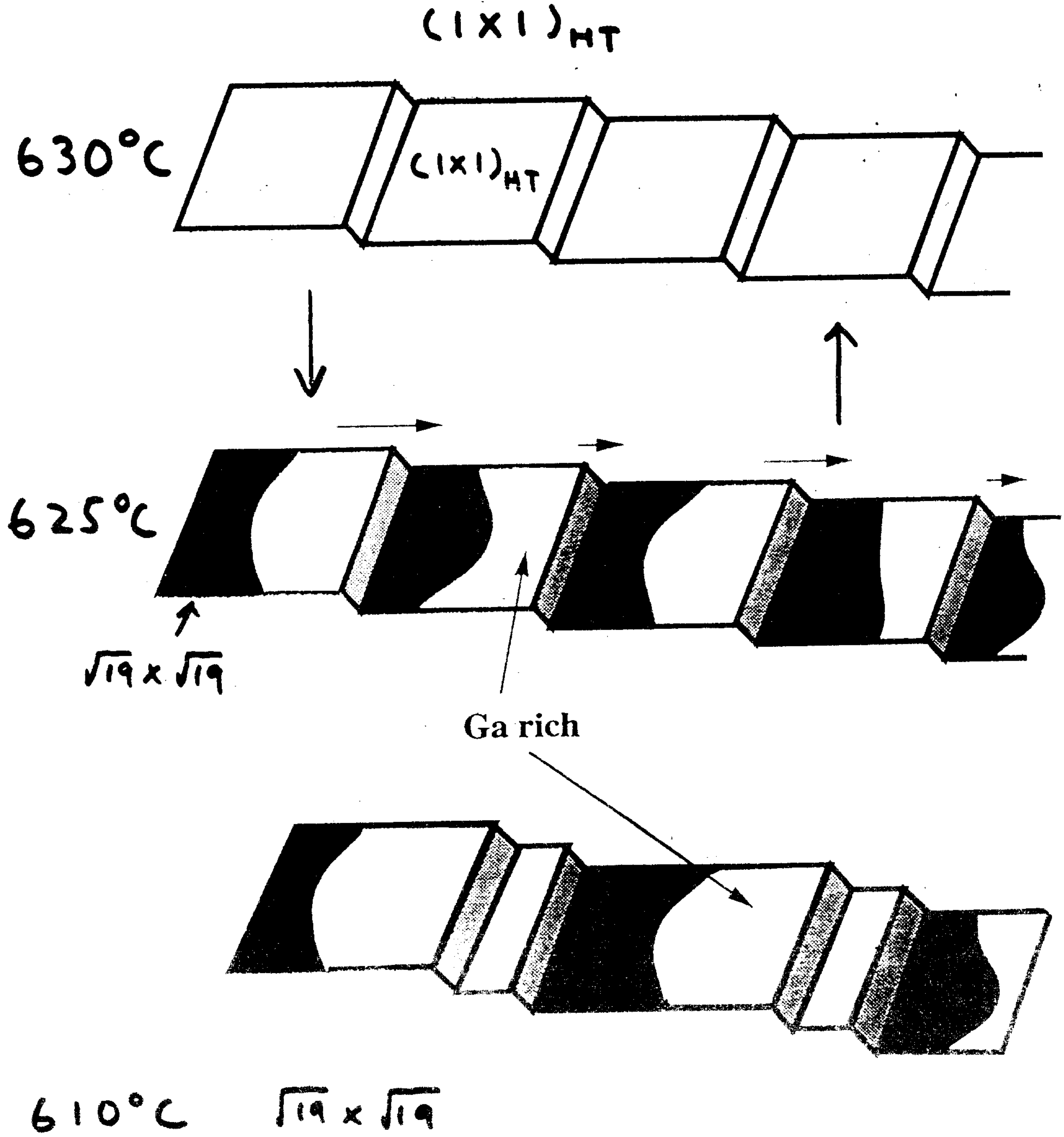
IMAGE
PROCESSOR
VTR

1. annealing

2. growth

$P_{As} = 2 \times 10^{-6}$ Torr



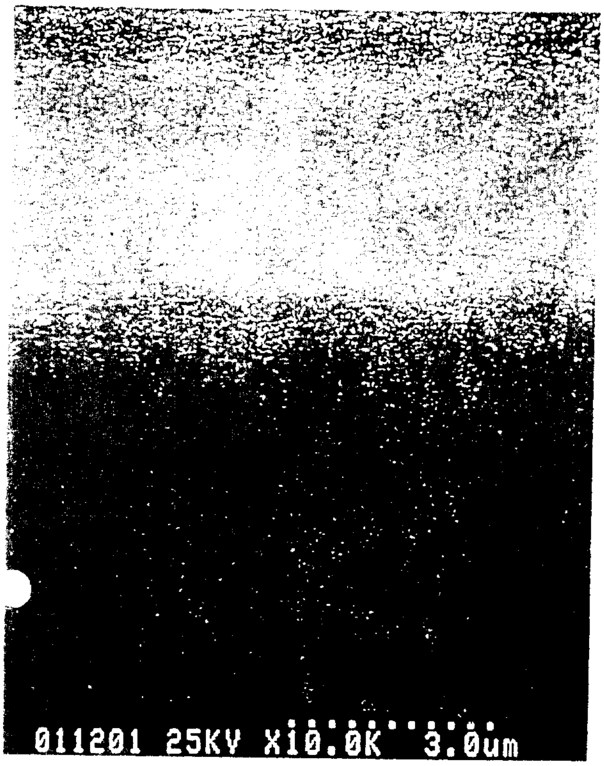


Fig. 2(a). Surface phase transition from $(1\times1)_{HT}$ to $\sqrt{19}\times\sqrt{19}$.

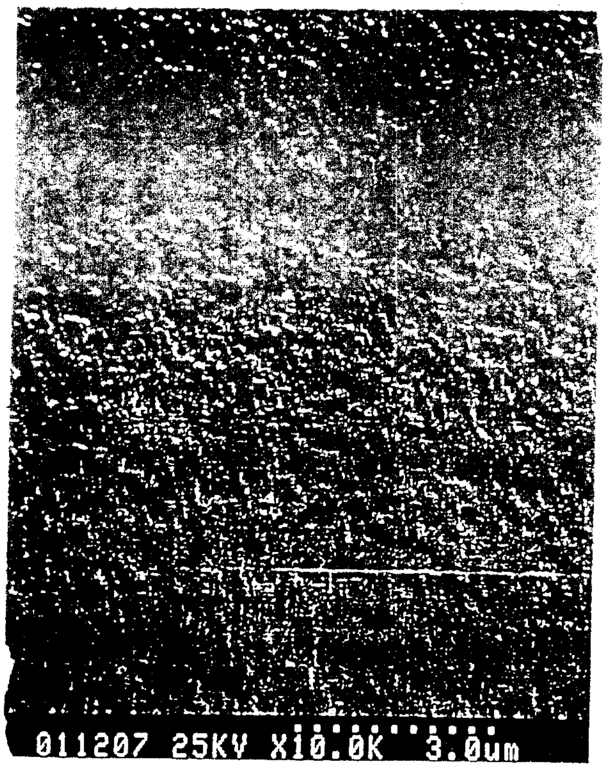
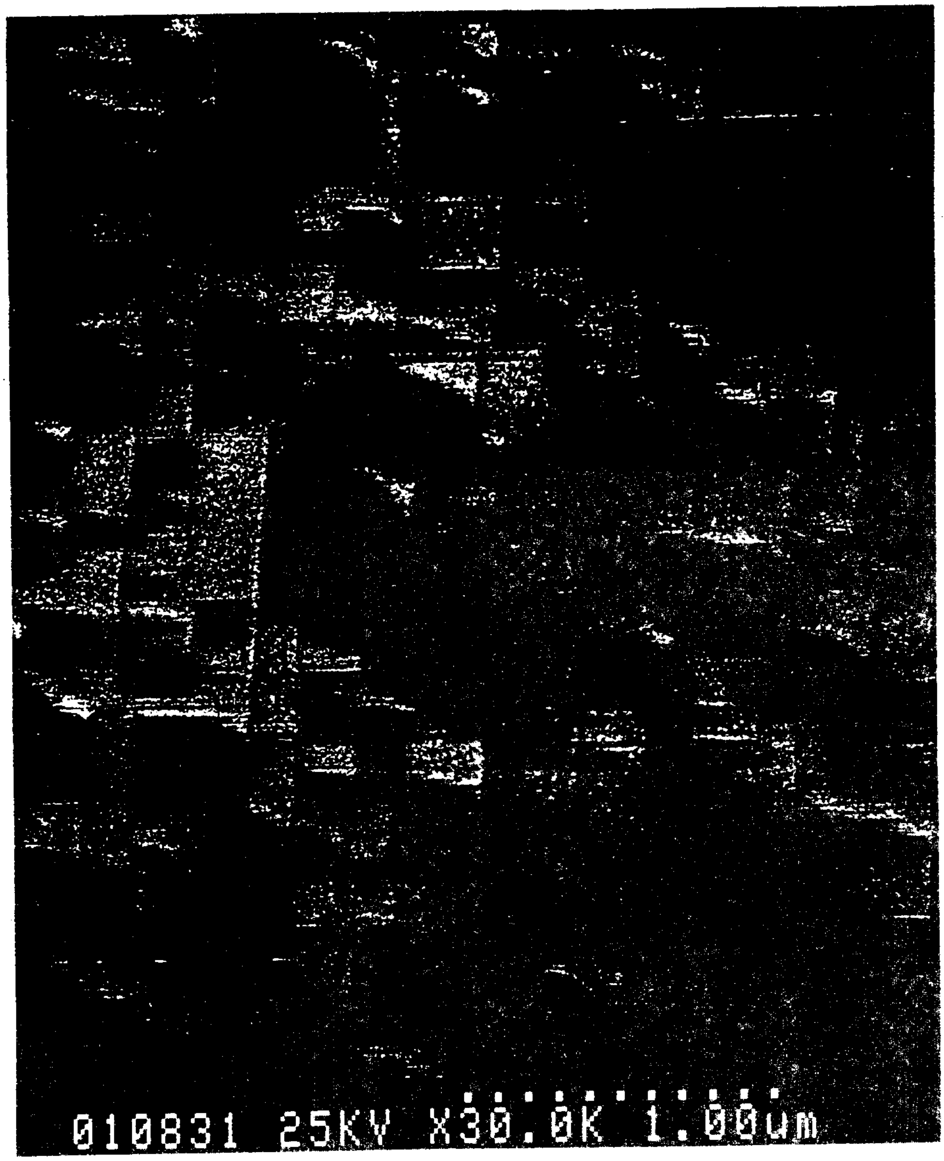


Fig. 2(b). Surface morphology in $\sqrt{19}\times\sqrt{19}$.

Fig. 3(a). Surface phase transition from $\sqrt{19}\times\sqrt{19}$ to $(1\times1)_{HT}$ slowly.

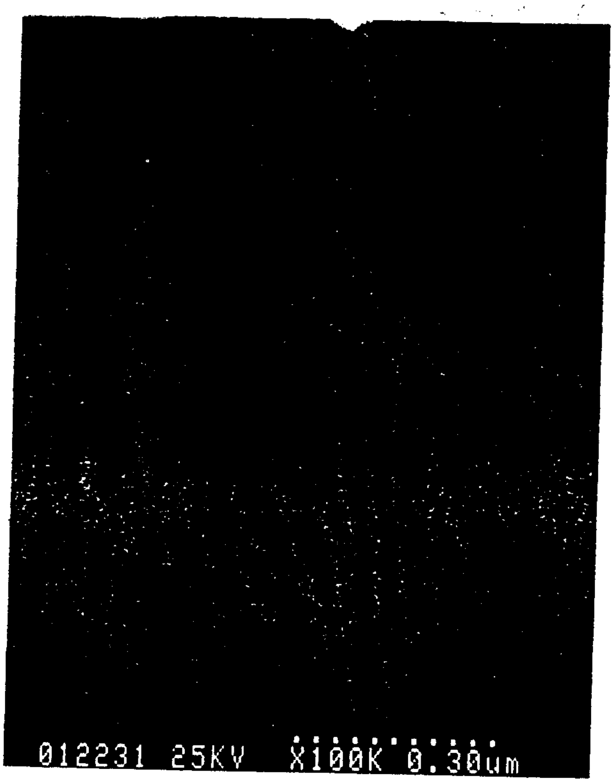
Fig. 3(b). Surface phase transition slowly from $\sqrt{19}\times\sqrt{19}$ and finally transforms to $(1\times1)_{HT}$ completely.



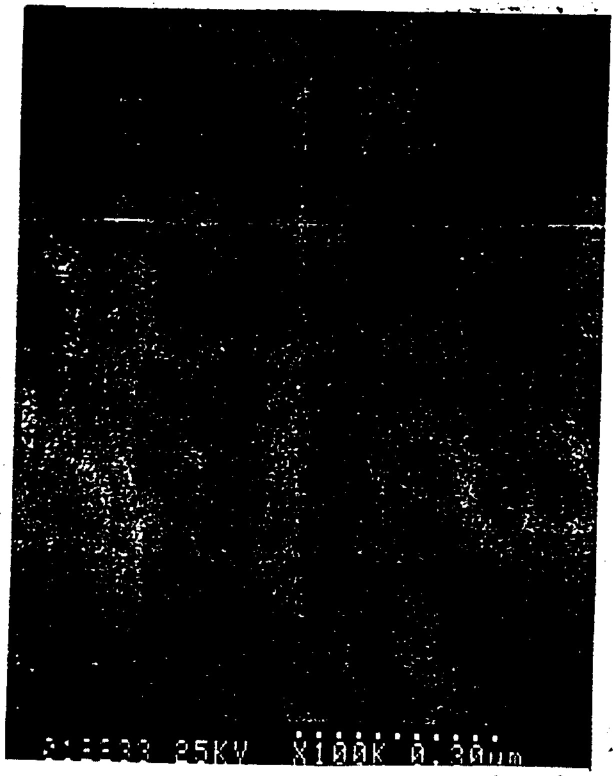
GaAs (111)B surface during the growth interruption at 625 oC, dark $\sqrt{19}$x$\sqrt{19}$ domains developed slowly from the step edges on to the terraces.

Effect of Growth temperature 620°C → 645°C

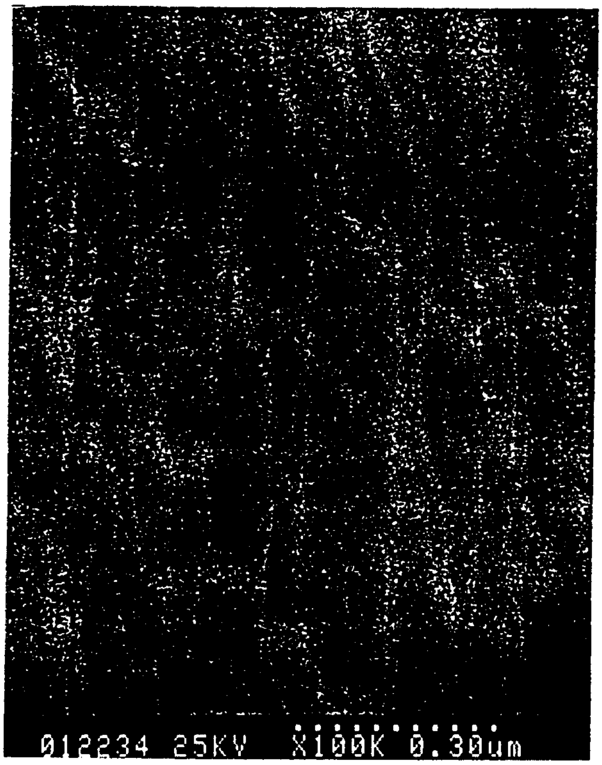
1-1



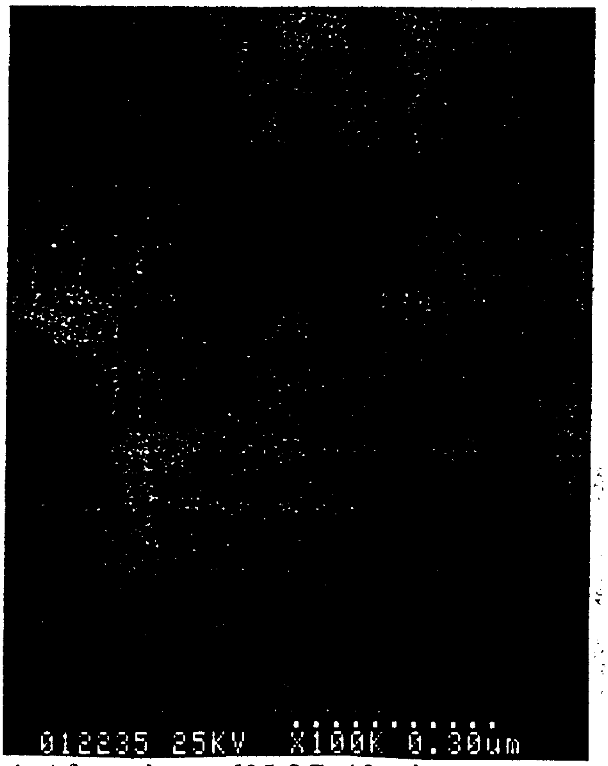
1. Growth at 620 °C, a Gr of 0.1 ML/s. just below the phase transition point. 0 min.



2. After raise to 625 °C, white 1x1 domain started to appear from the edges, and the macrostep spacing was growing. 4 min

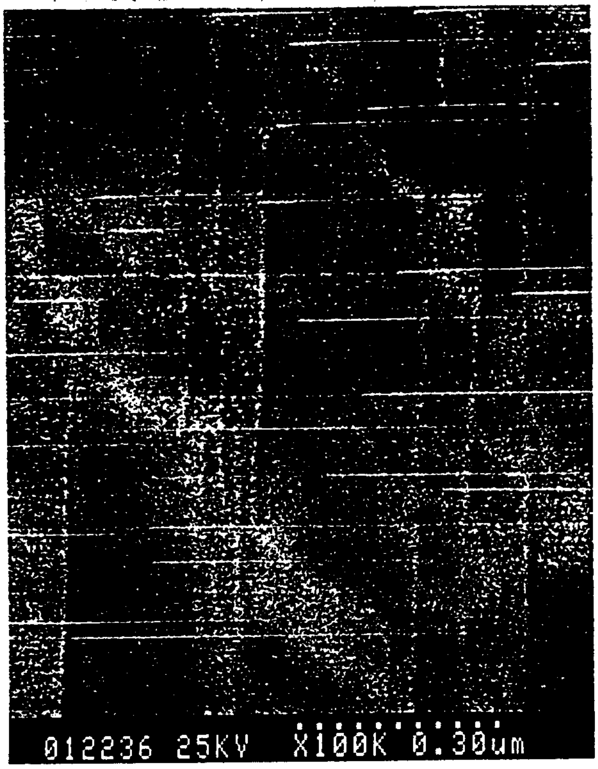


3. After raise to 630 °C, 6 min

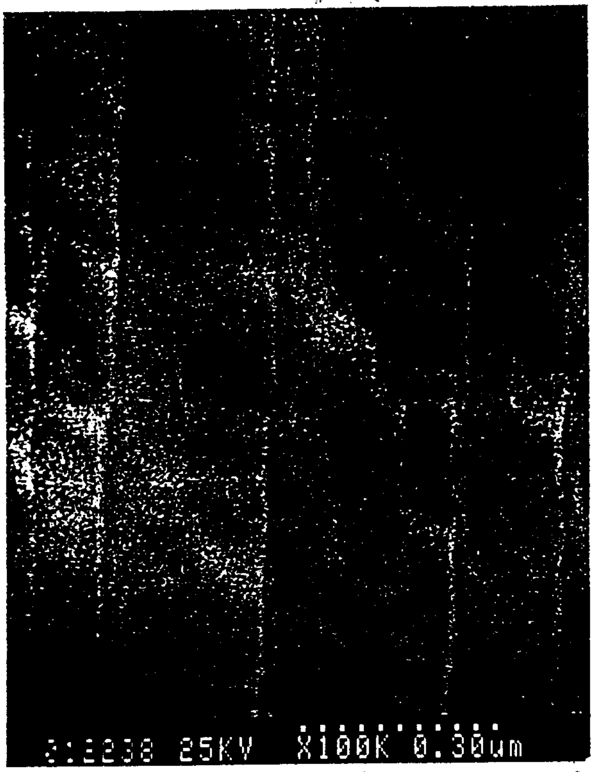


4. After raise to 635 °C, 12 min.

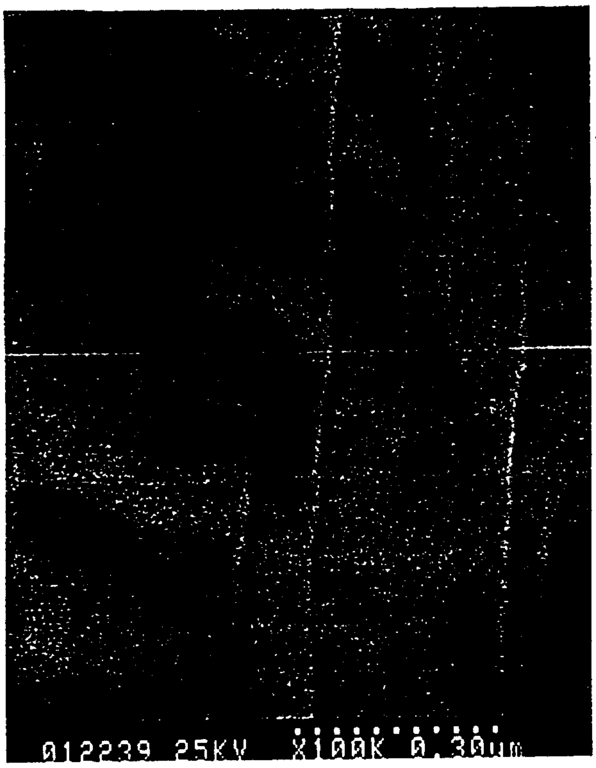
Enhance of step bunching 1-2



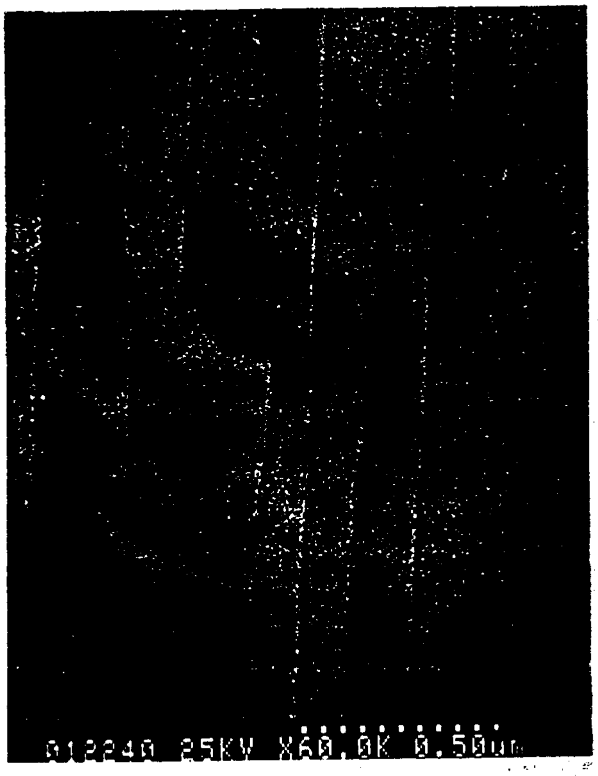
5. 18 min.



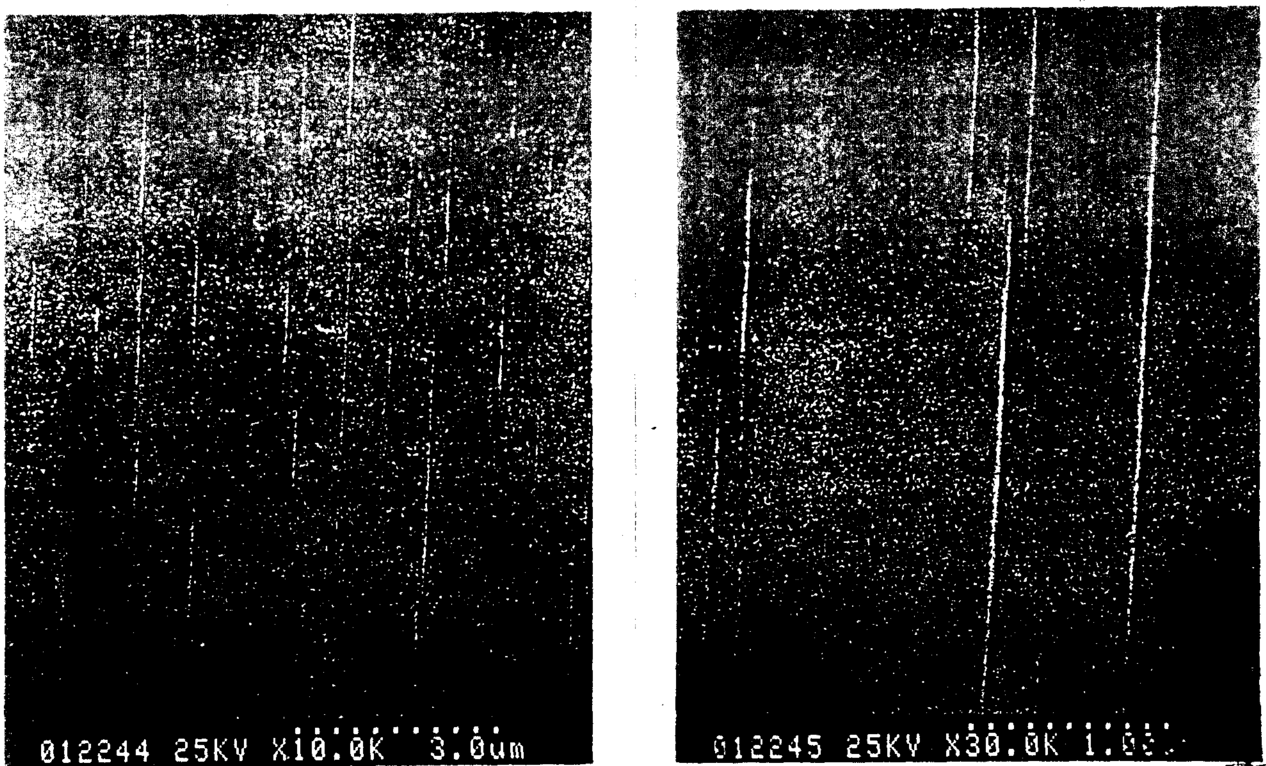
6. 23 min.



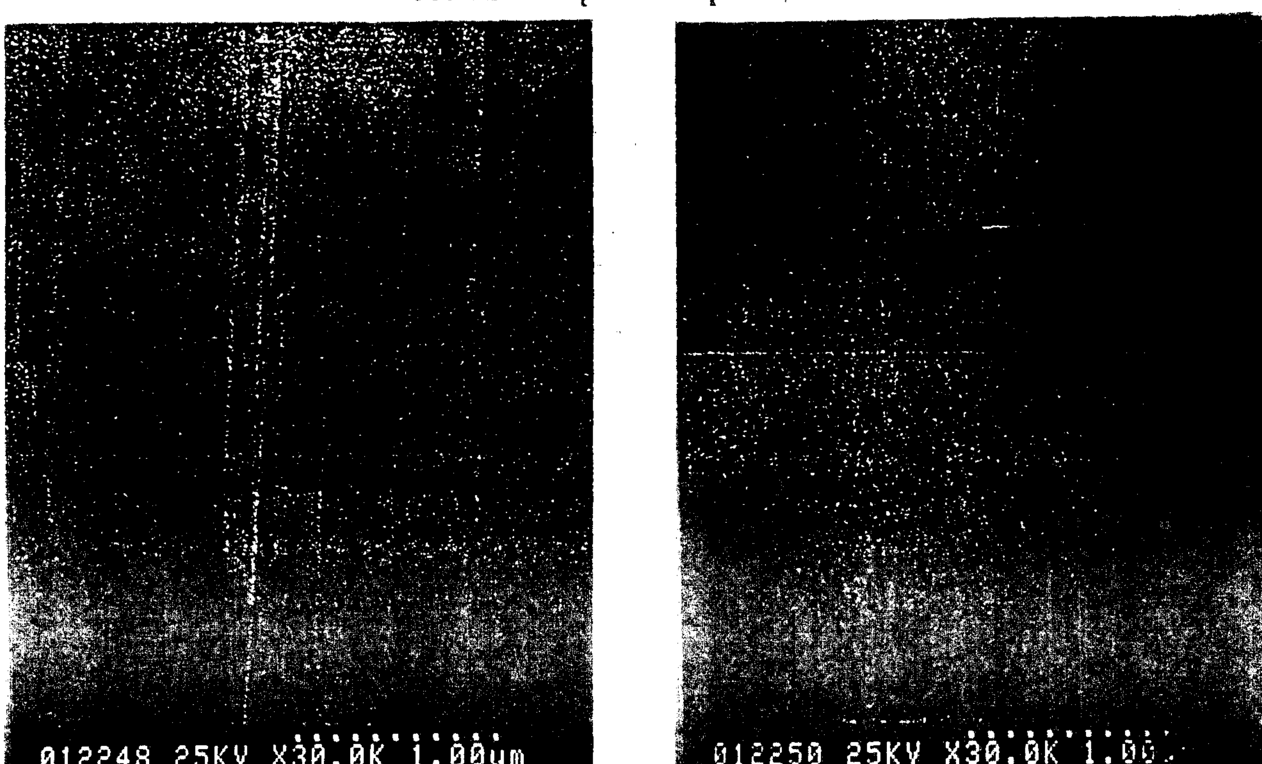
7. 30 min.



8. 34 min.

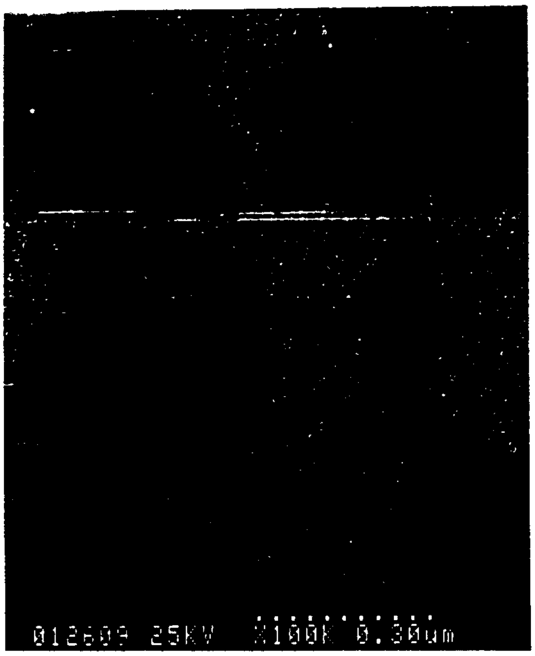


9. After slowly raised to 645 ^{o}C,the surface became complete 1x1 phase, 60 min.



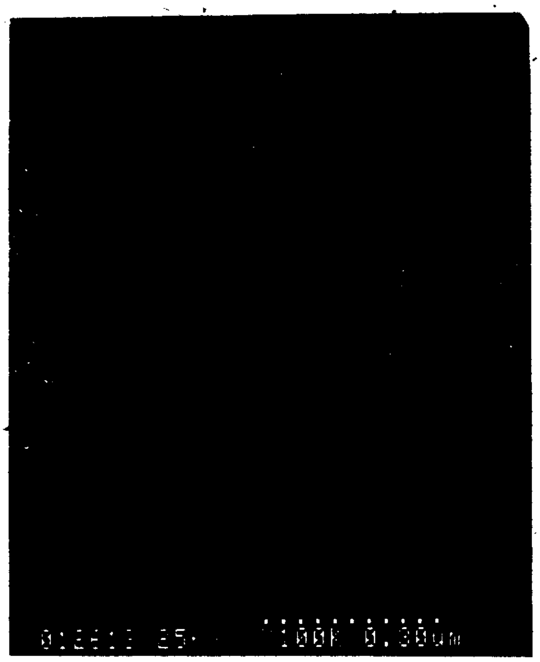
10. After lowered to 620 ^{o}C debunching

Effect of growth temperature 620 → 560 °C

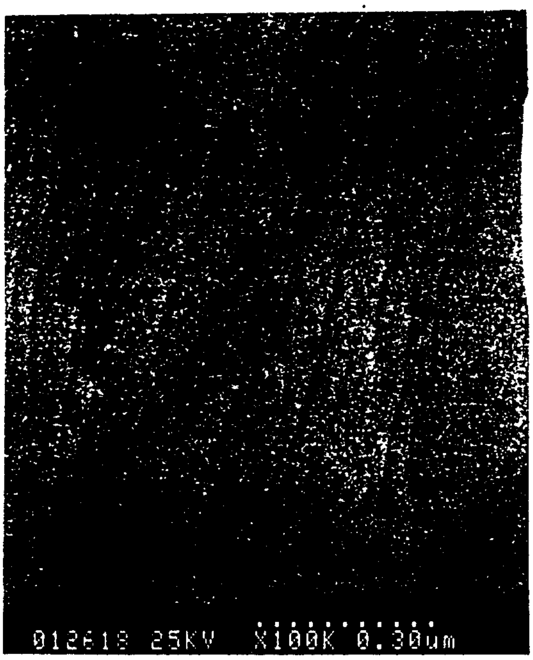


monolayer step

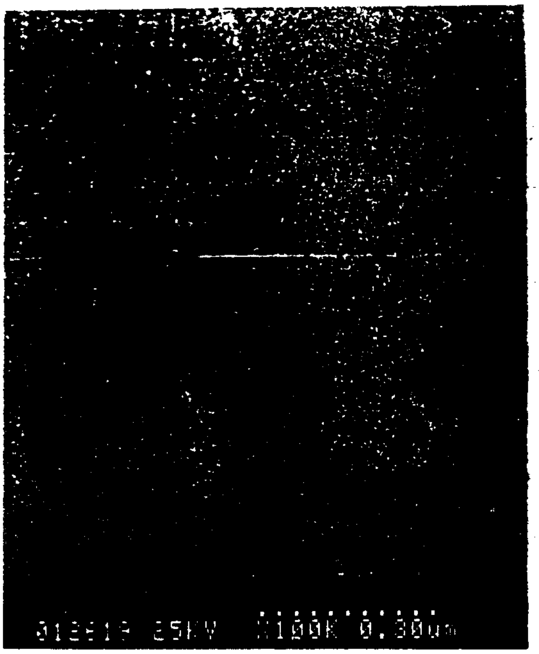
1. GaAs (111)B 1^{o} toward [110], MB growth at 620 ^{o}C, a Gr of 0.1 ML/s.



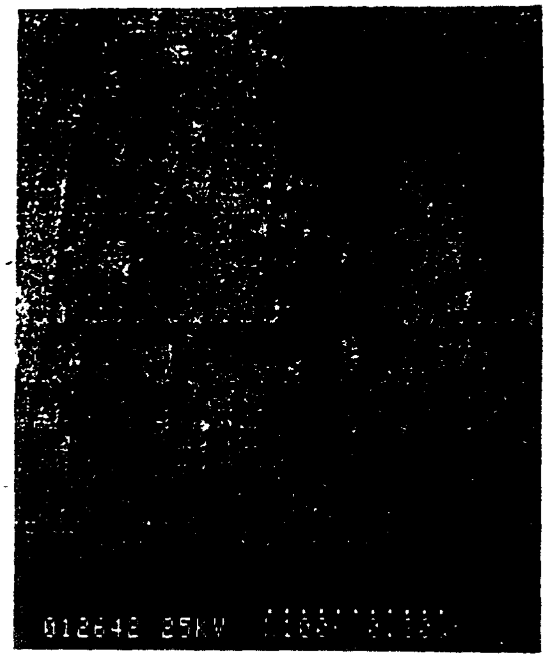
2. Growth at 610 ^{o}C.



3. Growth at 600 ^{o}c.



4. Growth at 590 ^{o}C.

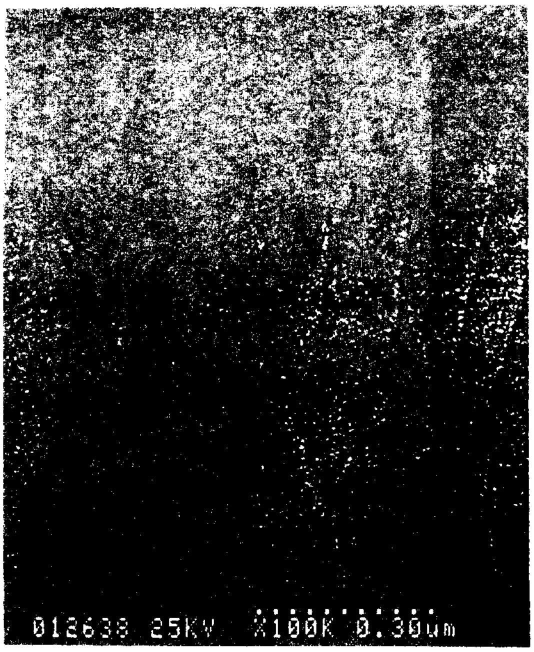


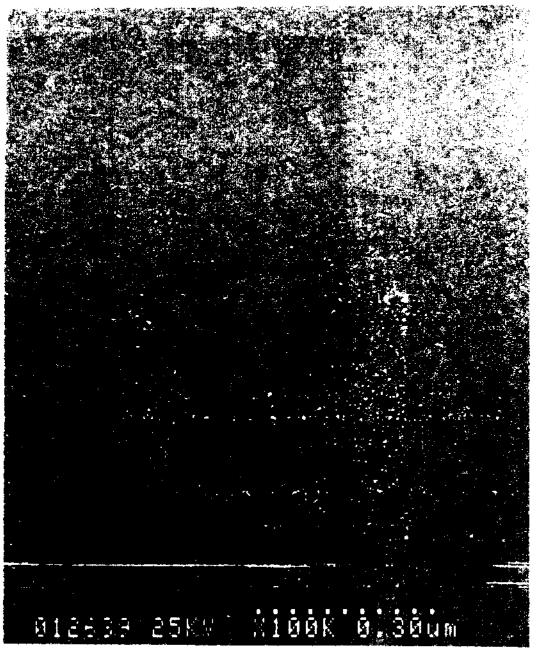
nearly equal step-distance

5. After stopped the growth for 5.5 min.

6. Wavy step shapes obtained when the growth was stopped at 620 ^{o}C.

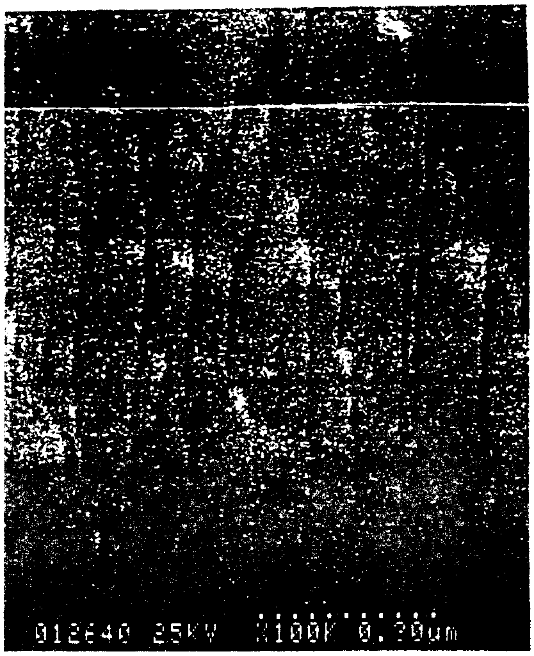
Effect of Low Temperature Annealing

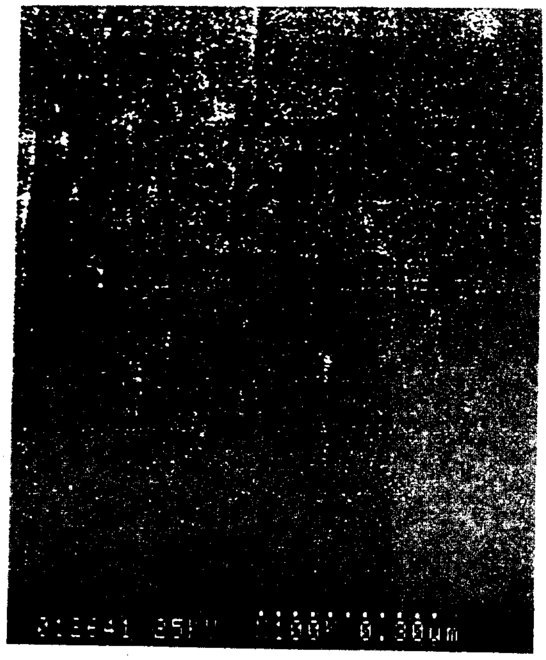




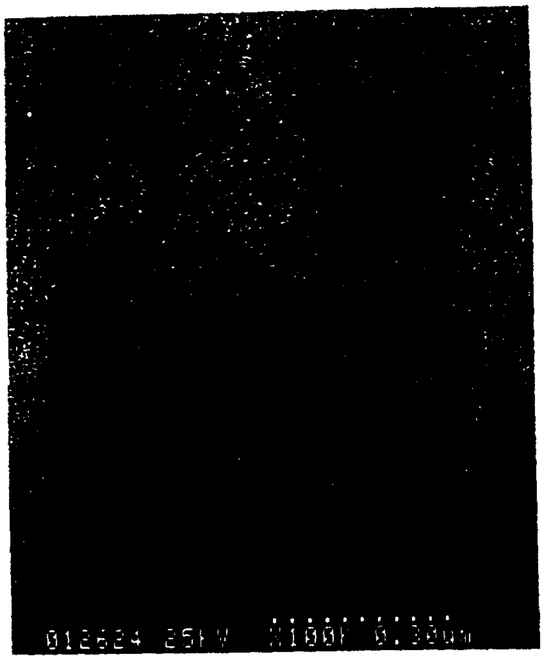
1. Growth at 560 ^{o}C, a Gr of 0.1 ML/s. Far away from phase transition points.
2. After stopped the growth for 1 min.

random inter-step distance

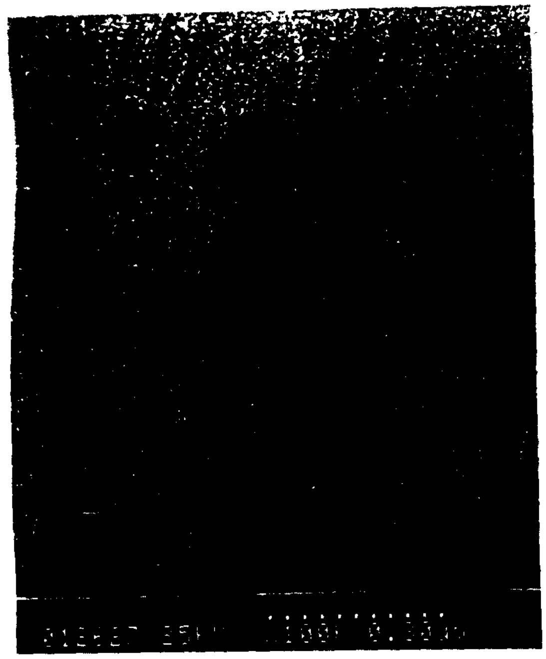




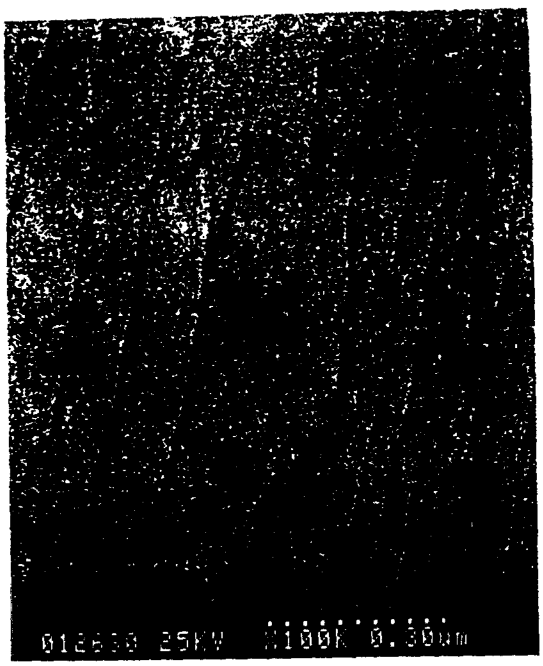
3. After stopped the growth for 2.5 min.
4. After stopped the growth for 4 min.



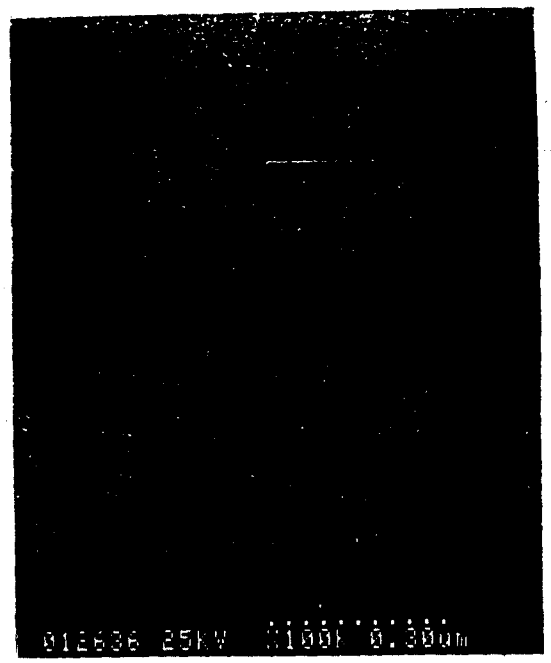
5. Continuous growth at 590 ^{o}C.



6. Growth at 580 ^{o}C.



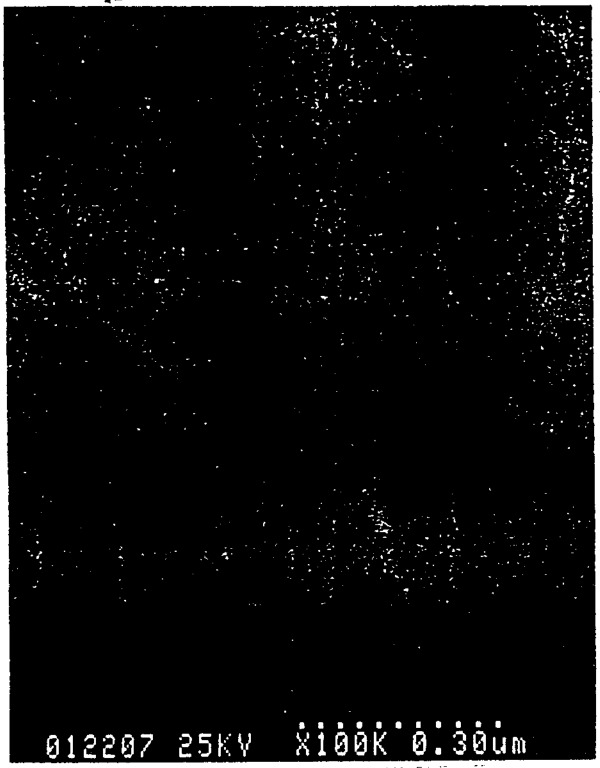
7. Growth at 570 ^{o}C.



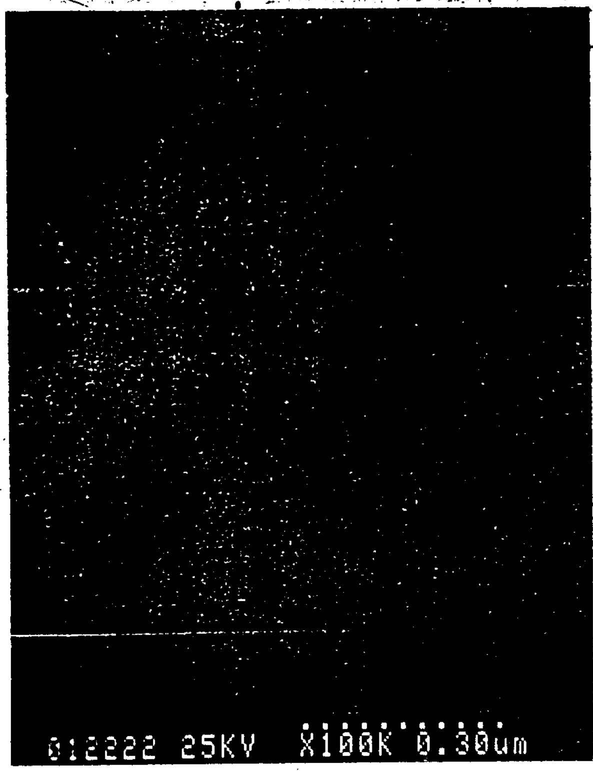
8. Growth at 560 ^{o}C.

macrostep (3~5 monolayer)

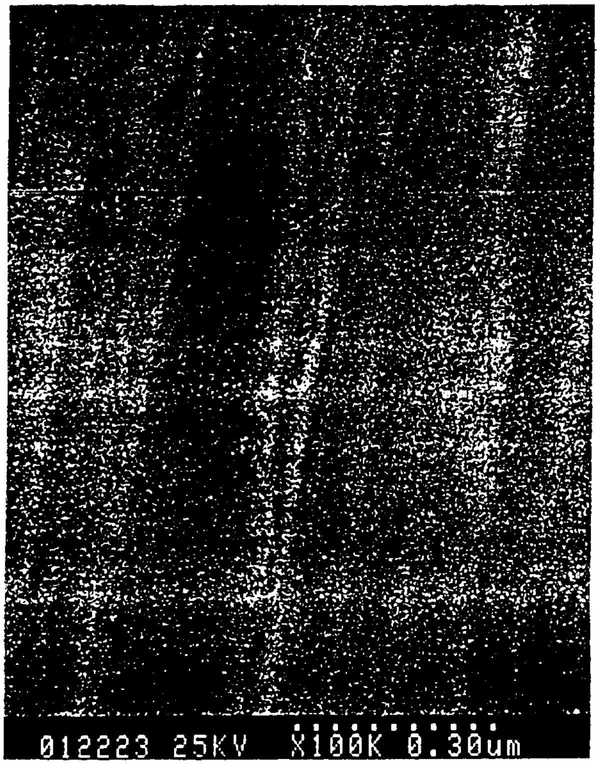
Effect of Growth Rate and Time of Annealing.



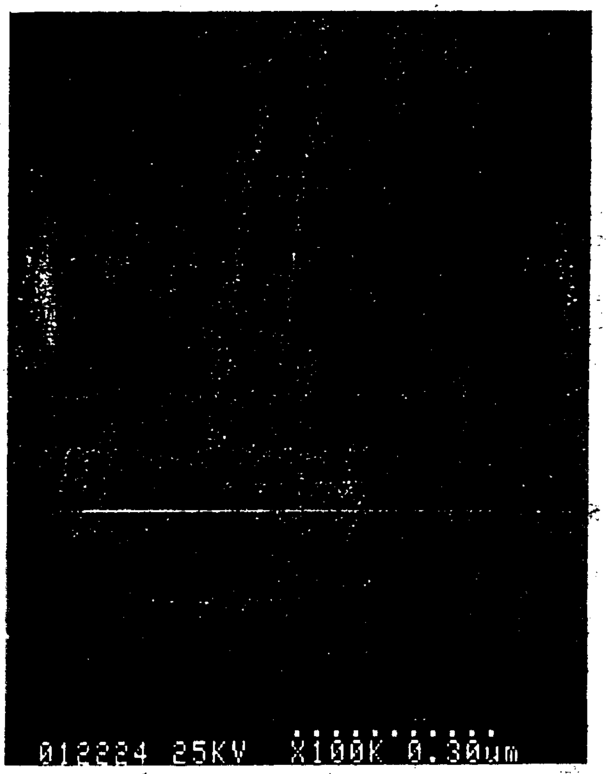
1. Growth at 620 ^{o}C, a growth rate (Gr) of 0.3 ML/s.



2. Growth at 620 ^{o}C, after Gr was changed to 0.1 ML/s.

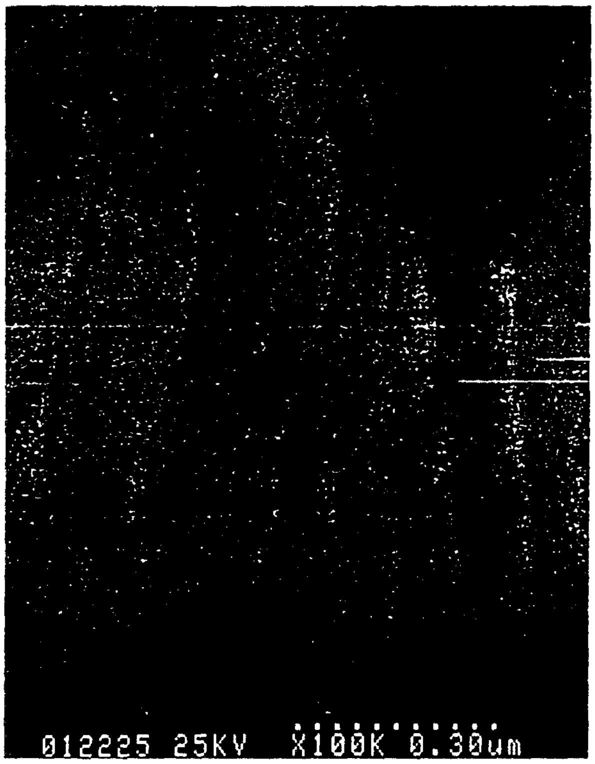


3. After stopped the growth for 3 min. Two domains are observed.



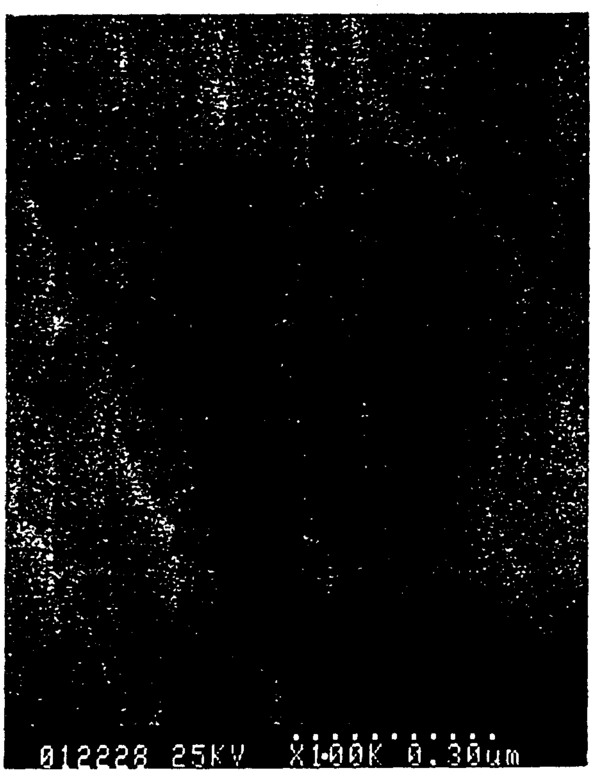
4. After stopped the growth for 6 min.

$(1\times1)_{HT}$: white $\sqrt{19}\times\sqrt{19}$: dark



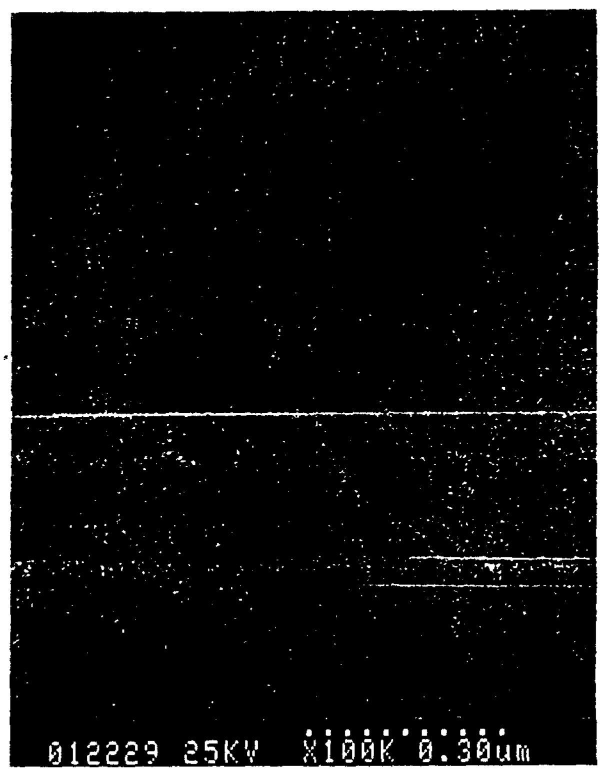
5. After stopped the growth for 8 min.

6. After stopped the growth for 13 min.



7. After stopped the growth for 15 min.

bunching



8. After restarted the growth for 1 min.

de bunching