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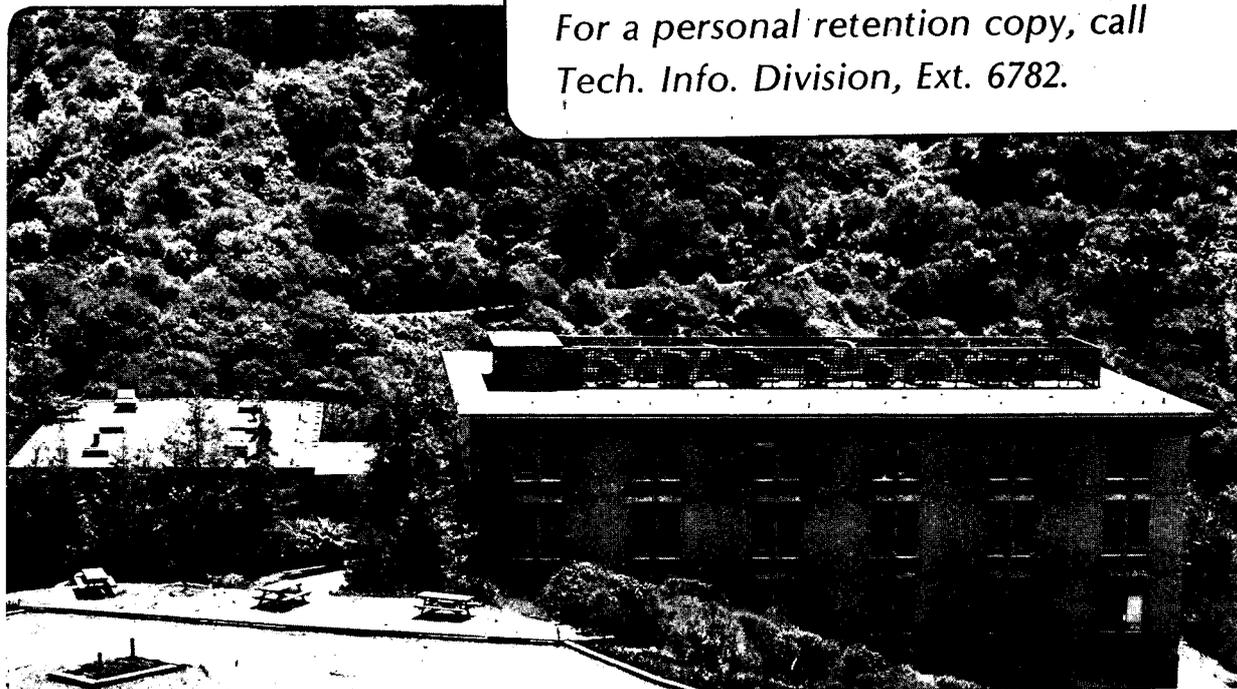
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ANNEALING OF INTERSTITIAL LOOPS IN ARSENIC IMPLANTED SILICON

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ABSTRACT

The annealing effect of different gas ambient (N_2 or O_2) on high dose (5×10^{15}) As ion implanted Si wafer has been investigated by using transmission electronic microscope. A two-layer defect structure is observed. The lower layer defects are interstitial type and attributed to the amorphous island below original crystalline-amorphous interface. The upper layer loops are As precipitation in the form of stacking fault. By comparing the growth/shrinkage rate of interstitial loops during neutral and oxygen annealing, it is proved that interstitial silicons are injected into bulk silicon during oxidation.

INTRODUCTION

When impurity atoms are introduced into crystalline silicon by ion implantation, knock on collisions between the energetic ions and lattice silicon atoms create Frenkel pairs and disordered zones, which during subsequent annealing form a variety of secondary defects: dislocation networks, loops, stacking faults and twins. The growth or annihilation of these defects can be profoundly affected by impurity/complexes and the annealing ambient. For example, when high dose implantations are done through screen-oxides into Si, high concentrations ($>10^{21} \text{ cm}^{-3}$) of recoil implanted oxygen are also incorporated into the amorphous regions. Subsequent annealing of such amorphous layers have been shown to cause the formation of numerous Si-O complexes which interact with the secondary defects to inhibit their growth or shrinkage.

The current practice for CMOS production involves implantation doses typically greater than $1 \times 10^{15} \text{ cm}^{-2}$ in order to achieve desired sheet resistance. This high dose implantation creates an amorphous layer which extends from the surface to about 2 Rp.

The post annealing of such a high dose implanted wafer involves the following processes: (i) regrowth of the amorphous layer, (ii) impurity redistribution, (iii) defect formation and evolution. This paper concerns mainly the last subject, although the other two processes are closely related.

For low temperature annealing, a two layer defect structure is frequently observed by TEM cross-section observation (Fig. 1b, 2b). Although similar defect depth distributions have been reported by Csepregi (1) and Sadana (2), their experiments involved high temperature implantation or low dose implantation respectively. In this paper the evolution of secondary defects is observed after room temperature or liquid N₂ temperature implantation of $>4 \times 10^{15}$ As followed by high temperature annealing for both O₂ and N₂ ambients. By comparing the growth/shrinkage rate of interstitial loops for neutral and oxidizing ambients information is also obtained concerning interstitial formation during oxidation.

EXPERIMENTAL

The experimental program was designed to be comparable to the general processing conditions of an all-implanted MOS device fabrication technology. The implantation was carried out into (001) Si wafers with As ion beams of 4 to 12 mA for doses 4 to 7×10^{15} cm⁻² at 100 or 120 Kev. Only the results of 5×10^{15} cm⁻² at 100 Kev have been described here. The wafers were either "bare Si" or with a 200Å thick oxide layer. The two sets of wafers were annealed at 1000°C with N₂ or O₂ or their combination. TEM was performed by using two-beam bright-field and weak-beam dark-field conditions to image the secondary defects in plan or cross-sectional view. Thin film annealing in an oxygen ambient was also carried out for comparison with the bulk annealing results.

RESULTS & DISCUSSION

(i) Formation mechanism of the lower layer of loops: During implantation, an amorphous band is created within the depth range where a critical ion dose is exceeded. The interface between this amorphous band and the still crystalline material is revealed in a TEM bright field cross-section as the boundary of the light area (Fig. 1a and 2a). The interface is not sharp, there are some disordered zones or amorphous islands inside the crystalline material near the interface.

During the initial stage of annealing, the amorphous material shrinks, solid state crystal growth consumes the amorphous band and the isolated amorphous islands. Near the lower amorphous-crystal interface in the vicinity of the isolated amorphous islands there is also an excess of interstitials: both dopant atoms and silicon. They cluster to form dislocation loops which subsequently grow in size by coalescing with neighboring loops and at the expense of smaller loops/point defects.

The amorphous band is constrained by the underlying crystalline material and is therefore in a state of biaxial compression. The crystalline material just below the amorphous band where the isolated amorphous islands exist is in a state of biaxial tension. Therefore, when amorphous islands regrow and simultaneously the excess interstitials cluster in this stress field, the resulting interstitial loops

preferentially have $1/2[011]$, $1/2[101]$, $1/2[0\bar{1}1]$, or $1/2[\bar{1}01]$ burgers vectors which tend to relax the stress. Loops with burgers vectors parallel to the surface were seldom seen, probably because their nucleation is not favored by the stress field. These loops in the lower layer, at least, after coarsening, were clearly identified as interstitial type by the method described by S. K. Maksimov (3).

(ii) Formation of Upper Layer Defects: As the crystal-amorphous interface starts to advance toward the surface, loops already formed at the lower layer act as sinks for additional interstitial atoms, no new loop nucleation takes place. The result is perfect crystalline regrowth for a short distance above the lower defect layer.

When the interface reaches the region corresponding to the peak of the as-implanted arsenic profile, a new defect formation mechanism appears to become important. At this region the arsenic concentration may be above its solid solubility in the crystalline material.

The maximum solubility of arsenic in silicon is $2 \times 10^{21} \text{ cm}^{-3}$ at 1100°C . (4) Although there is no available data for its solubility at 600°C , it is reasonable to expect that the solubility at 600°C is about an order of magnitude less, i.e., $2 \times 10^{20} \text{ cm}^{-3}$. The peak concentration of As in the as-implanted profile is $9.6 \times 10^{20} \text{ cm}^{-3}$ for $5 \times 10^{15} \text{ ion/cm}^2$ dose.

Therefore, arsenic atoms may be rejected in front of the advancing interface and could eventually form clusters which would be incorporated as precipitates, by using the TEM weak beam technique it was found that the clusters that are formed are small faulted loops with hexagonal shape about 200\AA in size.

On annealing the sample at high enough temperature, excess arsenic atoms should diffuse deeper into the bulk silicon, arsenic concentration in the implant-layer would fall below the solubility limit and the loops would shrink or evolve into ordinary dislocation loops. These defects in the upper layer do anneal out at 1000°C as shown in Fig. 1C. Another observation supporting the hypothesis that the defects are originally arsenic atom clusters is the fact that when annealed in an oxygen ambient coarsening of the lower layer of interstitial loops is accelerated while for the upper layer loops there is no effect (Fig. 4). This would be consistent with a high surface energy for the arsenic rich stacking fault inside the upper layer loops.

Similar upper layer defects appear for both bare and screen oxide implantation conditions, which suggests that oxygen is not important for their formation. However, the upper layer loops in through oxide implants are more resistant to annealing suggesting that pinning by oxygen complexes has increased their stability.

(iii) Evolution of Interstitial Loops: During annealing a reduction of the free energy of the system takes place in several ways. Larger loops are formed by coalescing with neighboring loops and by growing into larger loops at the expense of nearby smaller loops. Finally all loops shrink by loss of interstitial atoms to the surface. Image force can also cause some loops or parts of loops to glide to the surface.

As discussed above, the upper layer loops appear to be arsenic precipitates, they also should shrink during high temperature annealing due to loss of arsenic. Otherwise the evolution of loops in the upper layer is qualitatively similar to that for the lower layer.

During the $N_2/1000^\circ C/30$ min anneal in the "bare" implanted sample, interstitial loops apparently glided to the surface when their size reached about 3000\AA , (Fig. 1c). For the "screen oxide" implanted sample glide to the surface appeared to be inhibited by oxygen complex pinning (Fig. 3a). Even after extensive coarsening. The loops also had a more irregular shape suggesting oxide pinning (6). For $O_2/1000^\circ C/30$ min annealing, there were no loops remaining in the "bare" implanted sample, except a few long edge dislocation lines with $1/2$ (110) burgers vector parallel to the surface. This suggests that the excess interstitials injected by oxidation have accelerated the climb and coalescence of the lower layer interstitial loops. In the "screen oxide" implanted wafer, the coarsening of loops was also accelerated. Many loops reached 1μ in size before parts of the loop were lost by glide to the surfaces of the foil. Even these large loops had very irregular shapes (Fig. 3b).

The results of a thin film anneal in $O_2/1000^\circ C/5$ min is shown in Fig. 4. The same area is shown before and after the anneal. The small loops in the upper layer were unchanged and extensive climb, coalescence, and glide has taken place for the loops of the lower layer. This result confirms the behavior observed in bulk annealed samples for implants through a screen oxide. The upper layer loops are resistant to both growth and shrinkage while the lower layer loops are growing and coalescing due to injection of excess interstitials by oxidation at the surfaces of the foil.

CONCLUSION

- (i) Two layer secondary defect structures are formed during annealing after high dose arsenic implantation.
- (ii) The upper layer defects are arsenic clusters in the form of faulted $1/3$ $\langle 111 \rangle$ loops.
- (iii) The lower layer defects are interstitial $1/2$ $\langle 110 \rangle$ loops originating from the regrowth of amorphous islands and clustering of excess interstitials just below the amorphous-crystalline interface.

- (iv) Interstitial supersaturation results from annealing in an oxygen atmosphere that accelerates the growth of interstitial loops.
- (v) The injection of oxygen atoms during a through oxide implant results in pinning of dislocations that inhibits both growth and shrinkage of the secondary defects during annealing.

ACKNOWLEDGEMENTS

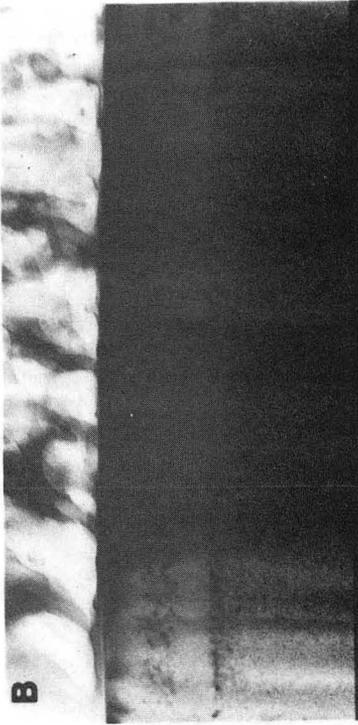
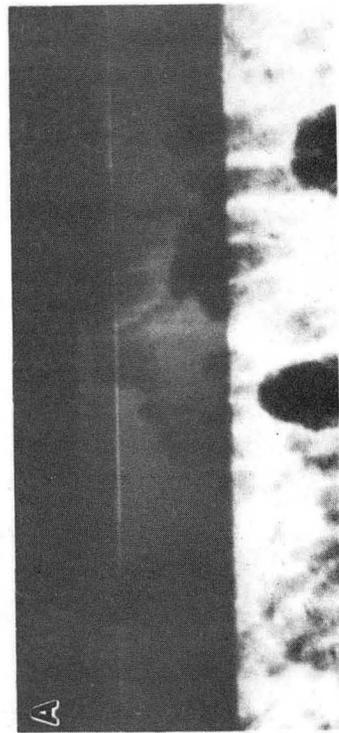
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REFERENCE

1. L. Csepregi, E. F. Kennedy, S. S. Lau, J. W. Mayer, and T. W. Sigmon, J. Appl. Phys. Vol. 29, 646 (1976).
2. D. K. Sadana, J. Washburn, and G. R. Booker, Phil. Mag. (b), Vol. 46, 611 (1982).
3. S. K. Maksimov, T. I. Lukyanchuk and M. M. Myshlyaev, Phys. Stat. Sol. (a) Vol. 24, 409 (1974).
4. F. Trumbore, Bell Syst. Tech. J., Vol. 39, 205 (1960).
5. D. K. Sadana, N. R. Wu, J. Washburn, M. I. Current, A. Morgan, D. Reed, and M. Maenpaa, Nucl. Inst. and Methods (in press).
6. S. M. Hu, J. Appl. Phys., Vol. 45, 1567 (1974).

Figure Captions

- Fig. 1 Cross-sectional TEM pictures of "bare" implanted Si wafer annealed in N_2 ambient. (XBB 834-3725)
- Fig. 2 Cross-sectional TEM pictures of through oxide implanted Si wafer with annealing condition: (a) as-implanted (b) $O_2/600^\circ C/10$ min (c) $O_2/1000^\circ C/10$ min (d) $O_2/1000^\circ C/30$ min. (XBB 835-3823)
- Fig. 3 Plane view TEM pictures of through oxide implanted Si wafer with post annealing ambient in either O_2 or N_2 ambients. (XBB 835-3824)
- Fig. 4. Thin film annealing of the same sample in O_2 ambient: (a) before annealing (b) after $O_2/1000^\circ C/5$ min annealing. (XBB 835-3822)

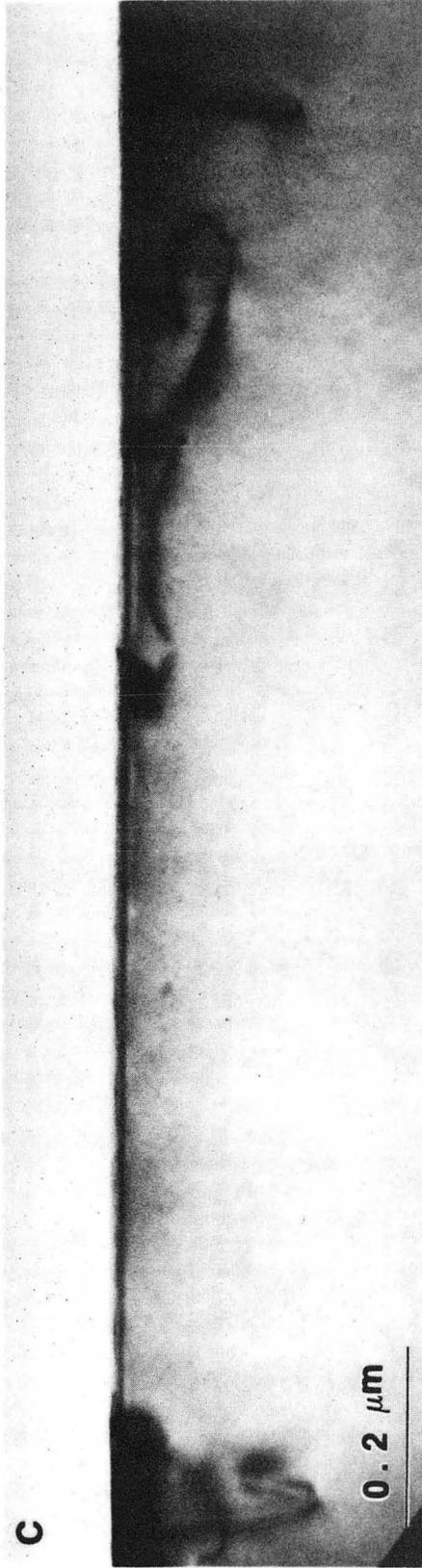


AS-IMPLANTED

$N_2/600^\circ C/20 \text{ min}$

7

C



$N_2/1000^\circ C/30 \text{ min}$

XBB 834-3725

Fig. 1

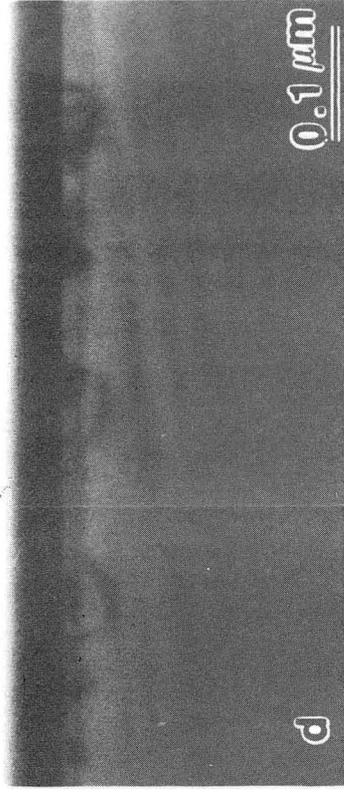
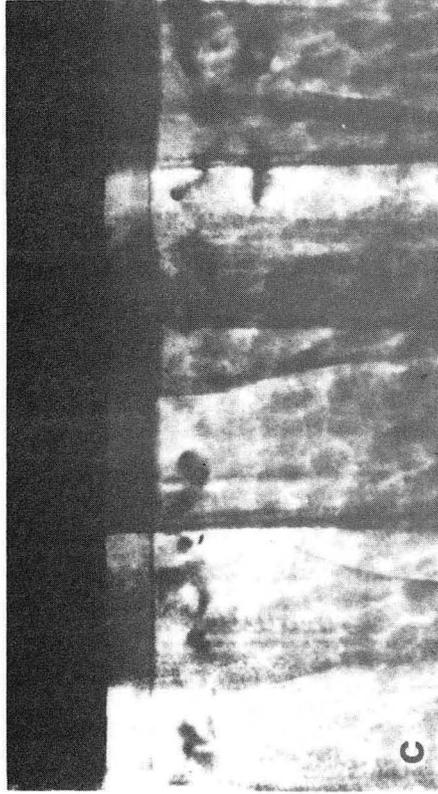
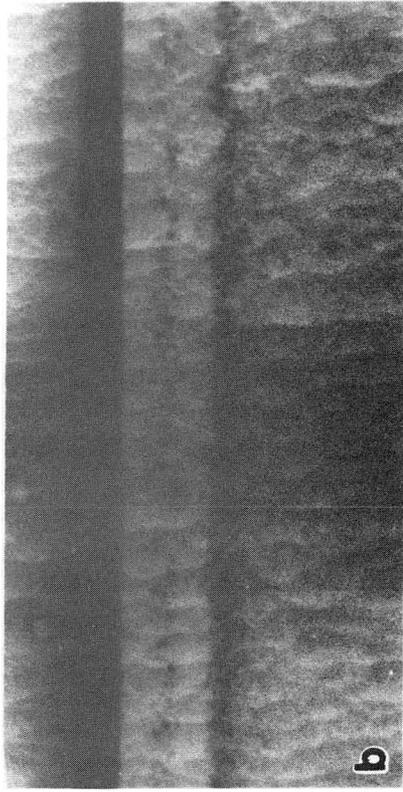
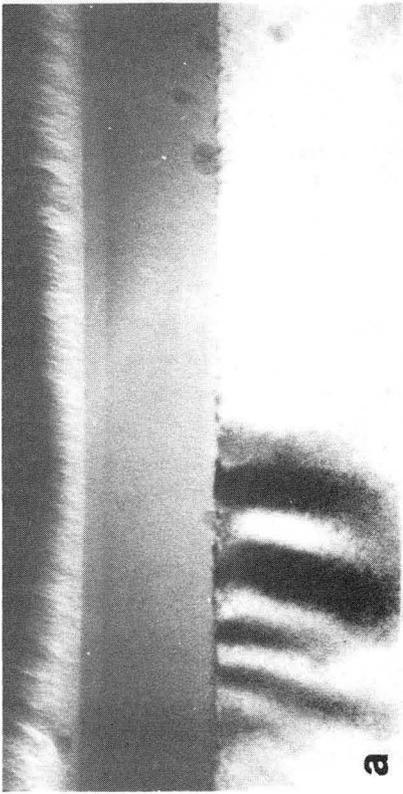
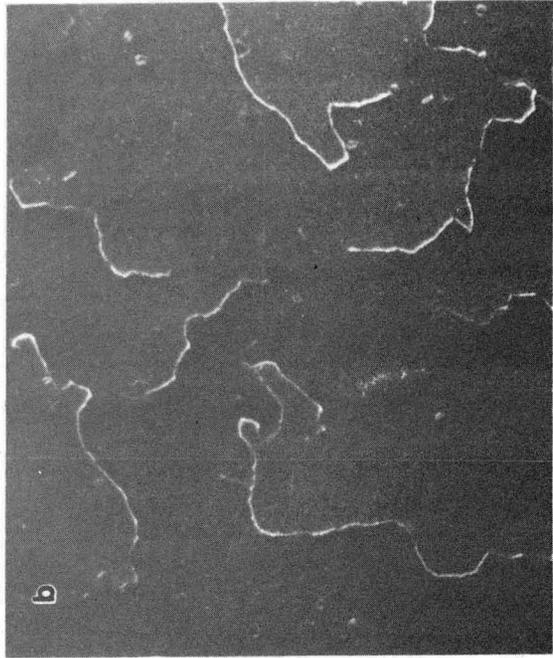


Fig. 2

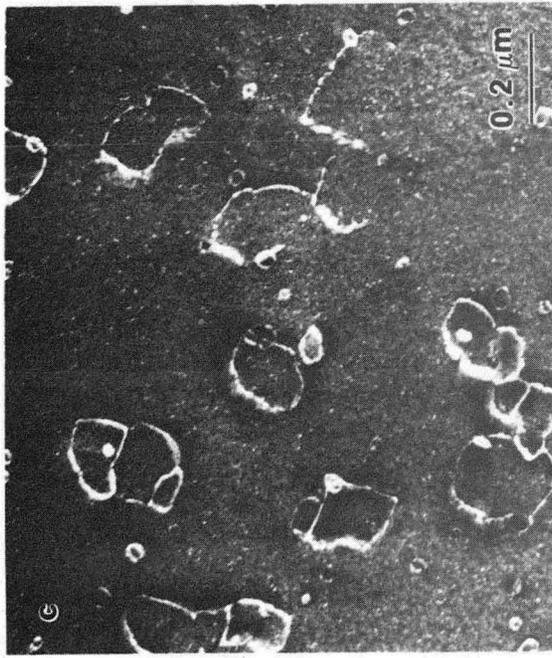
XBB 835-3823



$N_2/1000^\circ \text{ c}/30 \text{ min}$



$O_2/1000^\circ \text{ c}/30 \text{ min}$

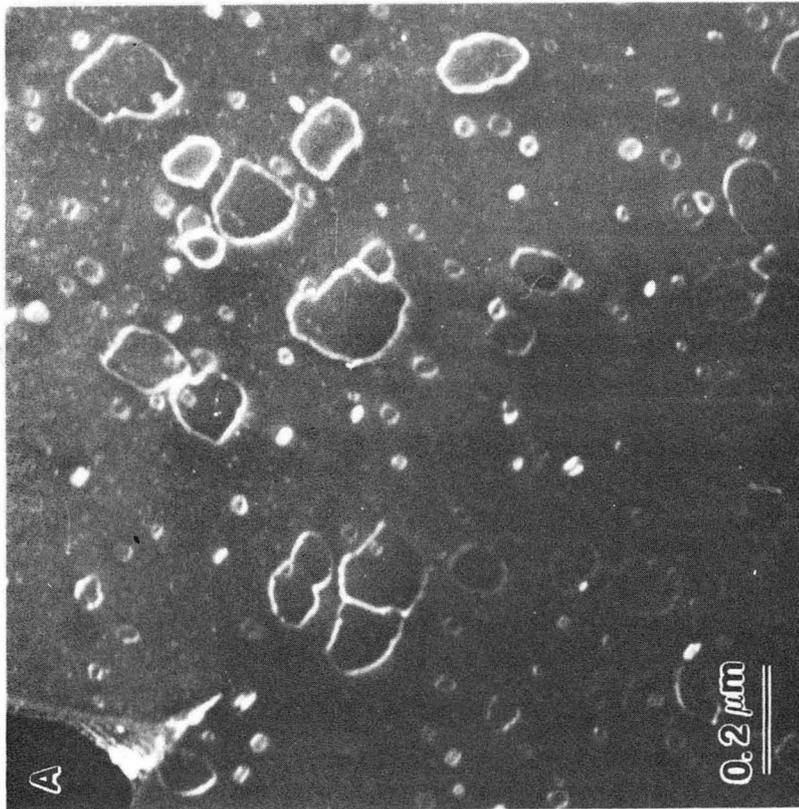
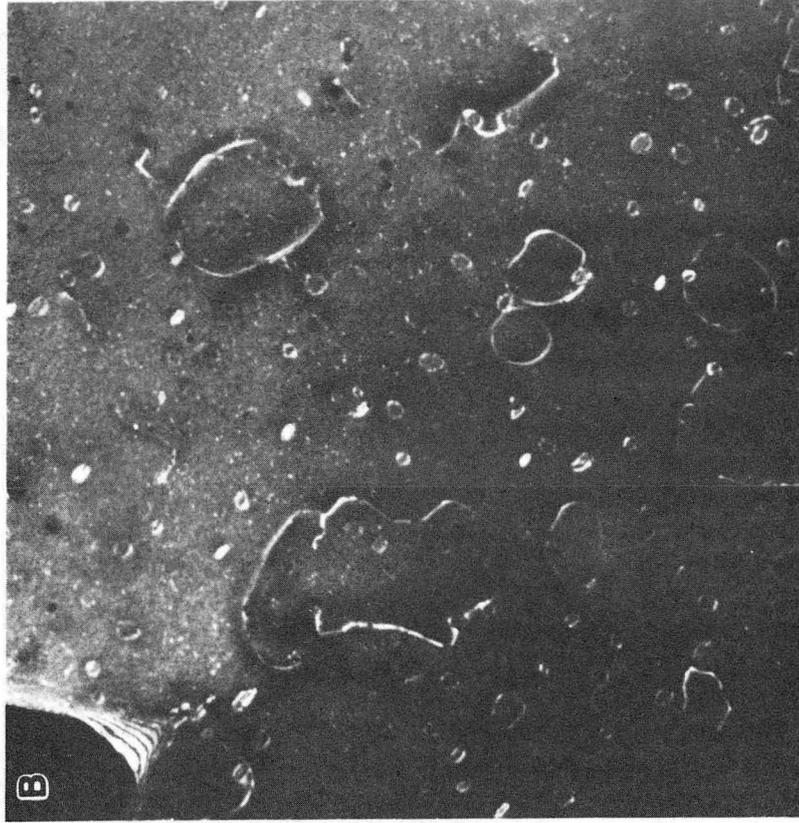


$N_2/1000^\circ \text{ c}/60 \text{ min}$



$N_2/1000^\circ \text{ c}/30 \text{ min} +$
 $O_2/1000^\circ \text{ c}/30 \text{ min}$

Fig. 3



XBB 835-3822

Fig. 4

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