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Effect of Heating Rate and Annealing Temperature on Twin Formation in As^+ Implanted ⟨111⟩ Silicon

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Abstract

The major secondary defect in post-annealed 5 x $10^{14}/cm^2$ As^+ implanted ⟨111⟩ silicon is microtwins. This was shown to be true for rapid thermal annealing similar to conventional furnace annealing. However, the twin density after rapid thermal annealing to 1100°C for 10 seconds was found to be greater than that in a comparable specimen furnace annealed at 1000°C for 10 minutes. Sheet resistivity after annealing was found to be closely related to the twin density of the specimen. Thermal stress was shown not to be the cause of this increase in twin growth. The higher average crystal regrowth temperature appears to be responsible for growth of more large area twin. Twins of small size were shown to anneal out at high temperature but large area twins did not. Twin free specimens were obtained by regrowth of the amorphous layer at low temperature which formed only small area twins followed by a high temperature anneal which annihilated all the small twins.

Introduction

Although rapid thermal annealing (RTA) was found to be very effective for avoidance of secondary defects in ion implanted ⟨100⟩ silicon[1], it has not been shown to be similarly effective for other orientation. Post-annealed ⟨111⟩ implanted silicon is known to have high residual disorder [2,3]. Microtwins and their accompanying boundary dislocations constitute the major defects [4,5]. However the factors affecting the size and density of microtwins in regrown (111) silicon wafers have not been clarified. Rapid thermal annealing would be expected to differ from conventional furnace annealing in at least two respects : The rapid heating rate, 200°C to 1100°C in 6 seconds, will result in regrowth of crystal into amorphous layer at a higher average temperature and consequently at a higher rate. Also thermal stress during heating should be greater than is the case for conventional furnance annealing.

In the present work heat pulse annealing was used to study the effect of heating rate and regrowth temperature on twin formation and growth in (111) As^+ implanted silicon.

Experimental

The 4° off ⟨111⟩ p-type boron doped 7-15Ω-cm Cz silicon wafers were implanted with As^+ 5 x 10^{14} $/cm^2$, 100 KeV at room temperature. Some of the specimen were implanted with 190 KeV to produce a thicker amorphous layer. The specimens were annealed in a ultra high purity argon atmosphere using 210T machine manufactured by AG Associates. Annealing was performed with various heating rates and heating cycles. A schematic representation of annealing cycles is shown in figure 1. Conventional tube furnace annealing with N_2 flow was used to compare with RTA.

The specimens were chemically etched with (HF + HNO_3) to prepare TEM thin foil specimens. A cross-section TEM specimen was also prepared to show defect distribution with depth. Four point probe sheet resistivity measurement were performed after each heat treatment.

Result and Discussion

The microstruture of the specimen furnace annealed at 1000°C for 1o minutes is shown in figure 2a with [111] beam direction. Most of the secondary defects are microtwins on (111) planes, and all four possible (111) habits were observed. The microtwins that are parallel to the surface can be seen to have hexagonal shapes with edges along ⟨110⟩ directions; some of the twins are isolated but many form clusters. Figure 2b shows the microstruture of a RTA specimen (1100°C for 30 sec.). The twin density is so high that almost all the twins intersect with each other forming complex clusters. Figure 3 shows the same specimens tilted to [123] to take the dark field image from a twin diffraction spot. The higher density of twins in the RTA specimen is again apparent (figure 3b). No change in twin density was observed after a second step annealing at 1000°C for 10 minutes. This indicates that the microstructure was relatively stable following its formation during crystal regrowth of the amorphous layer.

The calculated migration rate of an amorphous crystal interface in (111) silicon is

approximately 14Å/sec at 700°C and 200Å/sec at 800°C using the measured growth rate of $\dot{G}$ = 4.5Å/min at 550°C and an activation energy of 2.4eV. The thickness of the amorphous layer in 5 x 10^{14} As^+ 100KeV implanted silicon is about 1000Å. Therefore, the crystal regrowth has been completed even for the 1100°C heat pulse annealing before the highest temperature is reached. Compared with furnace annealing, the rapid thermal annealing has a faster heating rate, raising the wafer temperature from 500°C to 800°C in about 2 seconds, and from 800°C to 1000°C in another 2 seconds. Because of this rapid heating rate, the regrowth process in the RTA specimen would be expected to have occured at a higher average temperature and a faster rate. The increased thermal stress which also results from faster heating is another factor that might affect twin formation. To investigate the effect of this thermal stress, two implanted wafers were given a four step heat treatment with slow and fast heating and cooling rates. The first specimen (SHC) was heated at 2°C/sec from 400°C to 600°C, 650°C, 700°C and 750°C (four slow heating and cooling cycles) (figure 1a). The second specimen (FHC) was heated at about 100°C/sec from 400°C to 650°C, 700°C, 750°C and 800°C (four fast heating and cooling cycles) (figure 1b). The TEM result shows little difference in microtwin density for the two specimens (figure 4a, b); both have high twin density.

A TEM cross section specimen was prepared to study the depth distribution of defects in the specimen of figure 4a (figure 4c). Two variants of (111) habit twins appear edge-on in this [110] beam direction. Twins of large, medium and small size are present from the surface down to the depth of 1400Å, which was the thickness of the amorphous layer for a 190 KeV implantation. The small twins are at the greatest depth and twin size increases closer to the surface. This suggests that small twins are formed during regrowth of the partially amorphous materials in the transition zone between fully amorphous and crystalline materials , or during regrowth at the lowest temperature, and that large area microtwins are formed during regrowth of the fully amorphous layer which will occur at higher temperature in a contiuously heated specimen. Thermal stress during heating appeared not to have any influence on twin formation.

During conventional furnace annealing regrowth of the amorphous layer takes place at a lower temperature than is the case for rapid thermal annealing (RTA) and the longer holding time at higher temperature eliminates the smaller microtwins and dislocation loops. Both of these differences probably result in a lower twin population for the furnace annealed specimen compared to the RTA specimen. To further investigate the effect of regrowth temperature and high temperature twin annihilation, two step RTA was compared with a simulated furnace anneal (figure 1c,d). In both of these specimens regrowth of the amorphous zone would be expected to be completed at about 700°C and a 30 sec anneal at 1100°C would be expected to be comparable to more than 5 minutes at 1000°C. The microstructures are shown in figure 5a, c, and d. Both of these heat treatments produced microstructures and sheet resistivities comparable to conventional furnace annealing but in a shorter total time.

To investigate the effect of a still lower regrowth temperature a specimen was first annealed for 220 minutes at 550°C and then at 1100°C for 10 minutes. The microstructure before and after the 1100°C anneal are shown in figure 6a and b. Only the small microtwins and dislocation loops were formed and these were completely eliminated by the high temperature anneal. No twin diffraction spots were observed in figure 6c. This result suggests that even for conventional furnace annealing, the heating rate is too rapid through the critical 500°C to 600°C range to permit slow enough regrowth of the amorphous layer to avoid formation of large area twins. It is not yet clear why very slow regrowth at 550°C (4.5 Å /min) avoids formation of large area twins. However, it suggests that advance of the crystal-amorphous interface is less regular and that twins are always terminated by advance of another part of the crystal growth front ahead of the extending twin before it reaches a size of more than about .05 m. At higher temperatures and consequently higher growth velocity this does not happen and a microtwin once nucleated on an inclined habit plane frequently propagates all the way to the surface.

Table I summarizes the results and shows that measured sheet resistivity correlates with twin density.

Table I

specimens	FA1	RTA1	M1*	MH2*	FAS*	L1	LH2
heat treatment	1000°C 10min.	1100°C 10sec.	700°C 30sec.	700°C/30s & 1100°C/ 10sec.	furnace anneal simulation	550°C 220min.	550°C/220m & 1100°C/ 10min.
sheet (Ω/□) resisitivity	202	292	400	193	192		
maximum twin size	large	large	large	large	large	small	zero
twin density	medium	high	high	medium	medium	high	none

* specimens implanted with 190KeV.

Conclusions

1. Rapid thermal annealing of an As^+ implanted (111) silicon wafer results in a higher microtwin density than that resulting from conventional furnace annealing.
2. Higher thermal stresses associated with the higher heating rates during rapid thermal annealing are not the cause of the increase in twin density.
3. The higher microtwin density is caused by regrowth of the amorphous layer at a higher average temperature and therefore at a greater rate.
4. Very slow regrowth of the amorphous layer at 550°C results in formation of only small area microtwins, that are unstable at higher annealing temperature and are completely eliminated by an 1100°C anneal.
5. Sheet resistivity shows a correlation with microtwin density.

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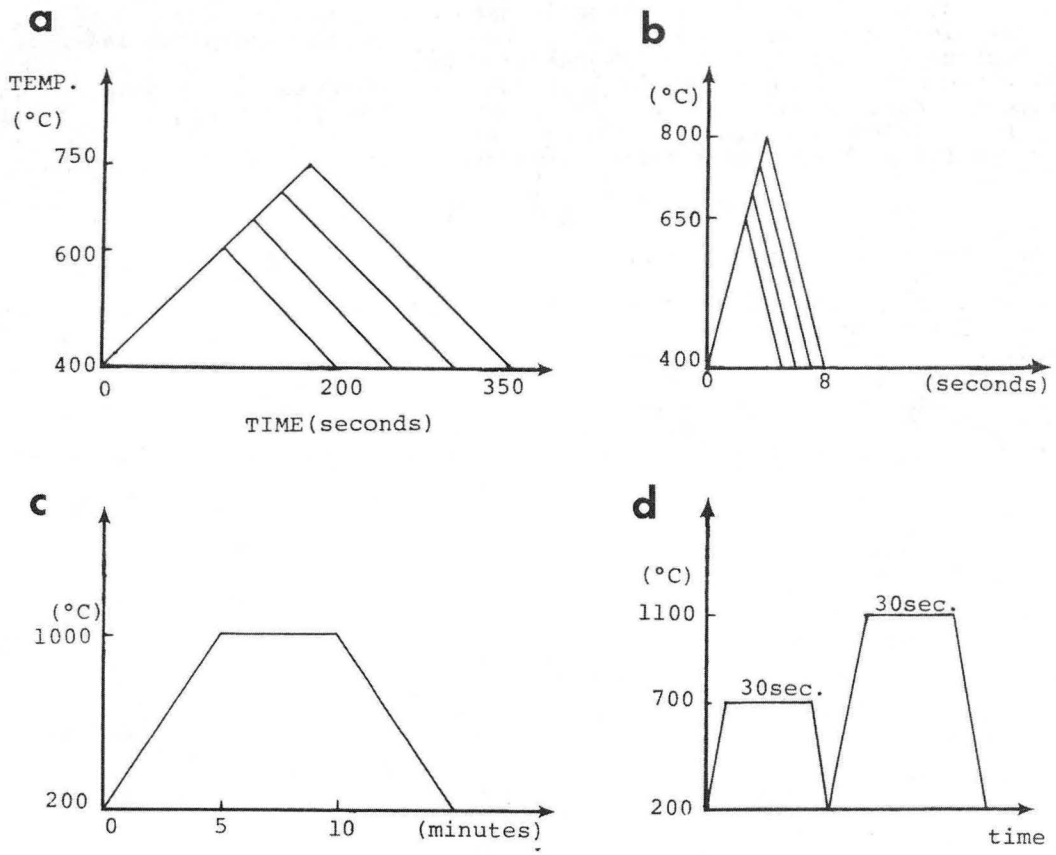


Figure 1. Schematic annealing process for: (a) SHC, slow heating cycle. (b) FHC, fast heating cycle. (c) FAS, furnace anneal simulation. (d) MH2, medium and high temperature two steps. (XBL 849-3978)

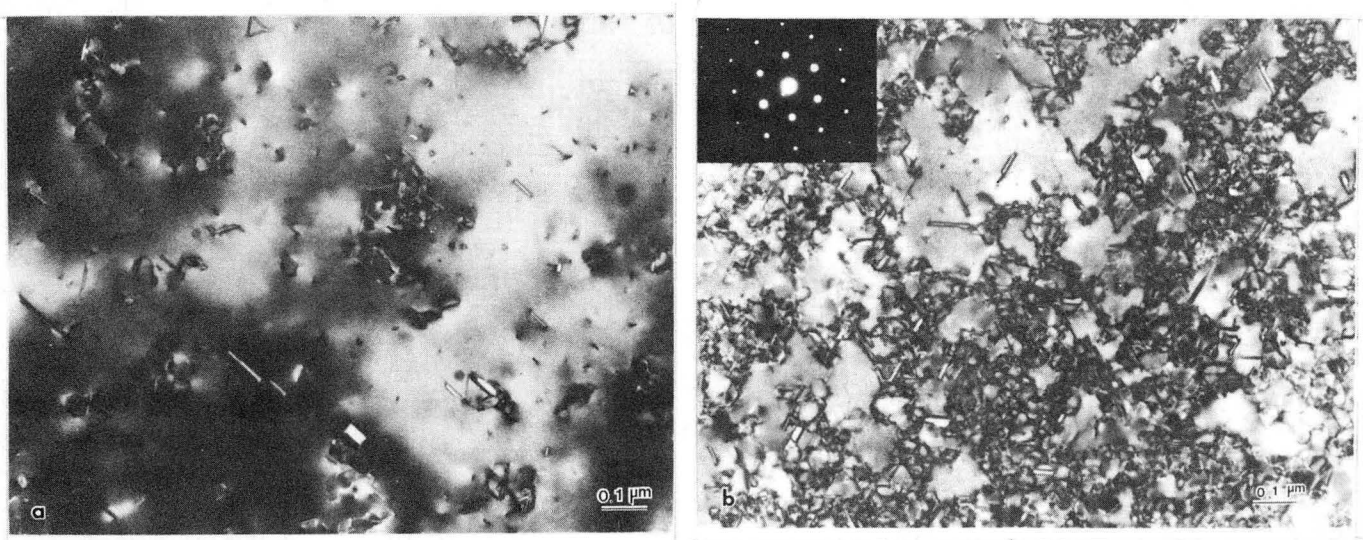


Figure 2. Bright field image in [111] beam direction : (a) 1000°C for 10 minutes furnace anneal. (b) rapid thermal annealing at 1100°C for 10 seconds. (XBB 841-267a)

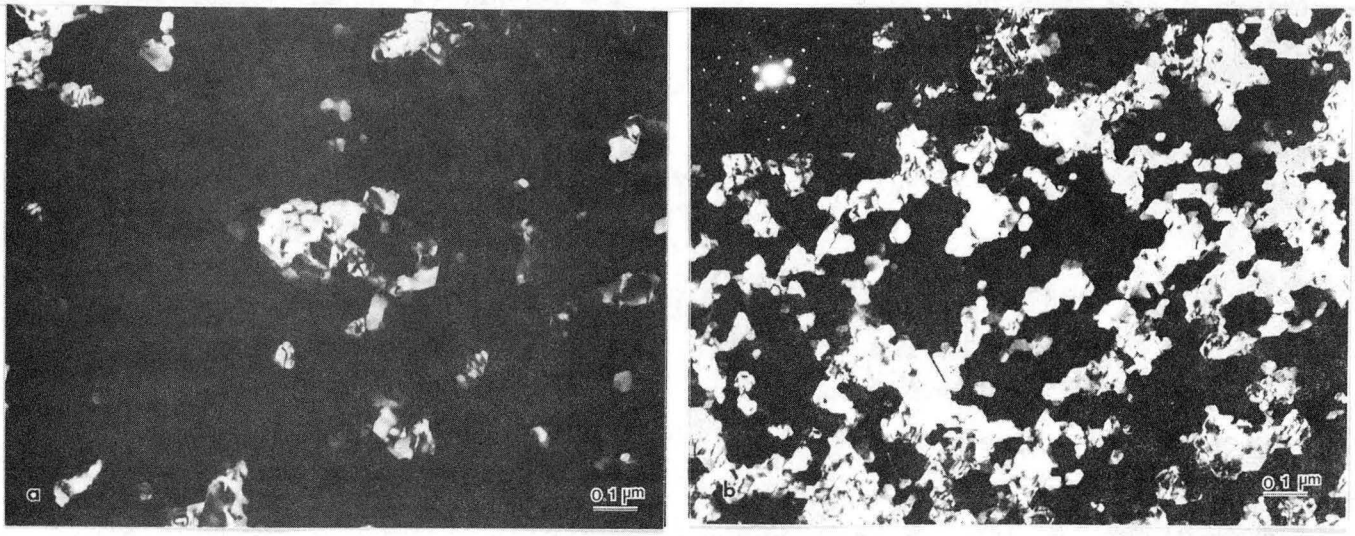


Figure 3. Dark field image taken from $\frac{1}{3}[\bar{5}11]$ twin spot in [123] beam direction: (a) furnace anneal at 1000°C for 10 minutes. (b) rapid thermal anneal at 1100°C for 10 seconds.

(XBB 841-268a)

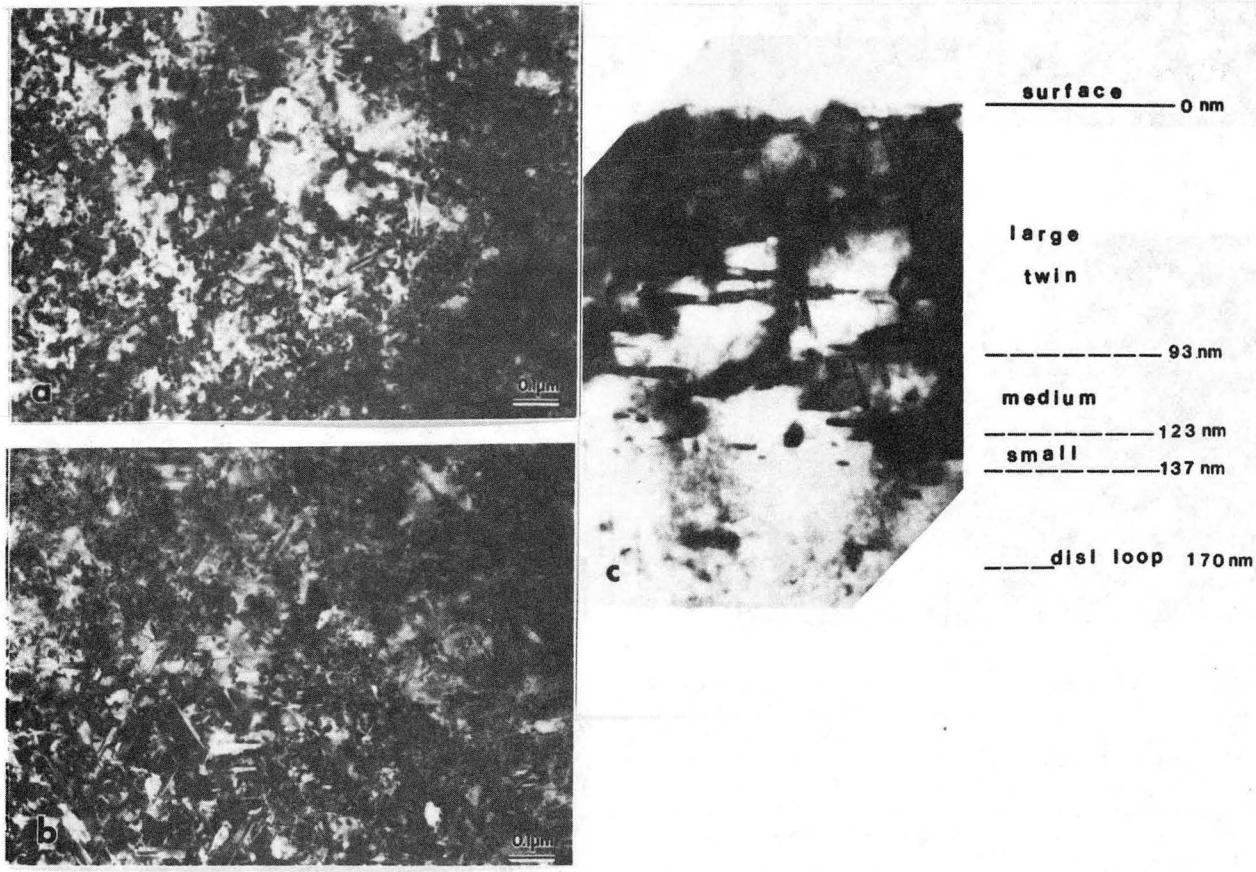
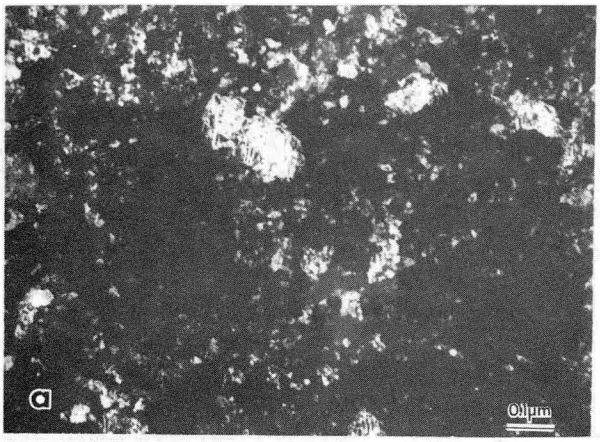
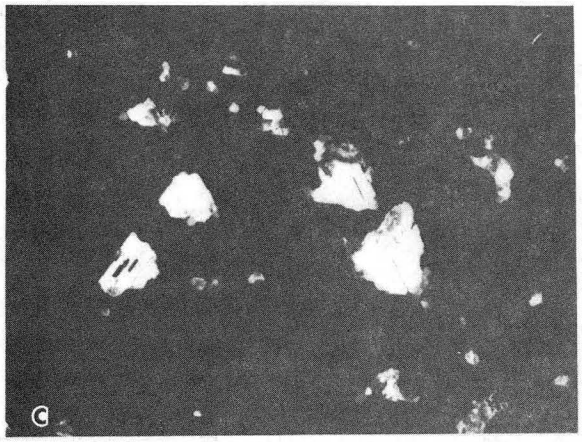


Figure 4. Bright field image in [111] beam direction for: (a) SHC specimen, (b) FHC specimen. (c) cross section microstructure of SHC in [110] beam direction shows the depth distribution of defects.

(XBB 841-266a)





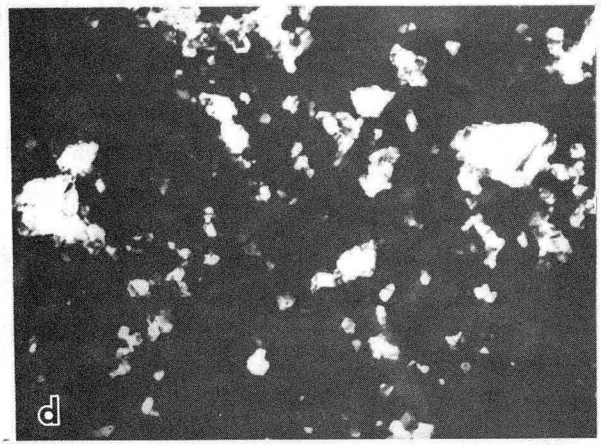
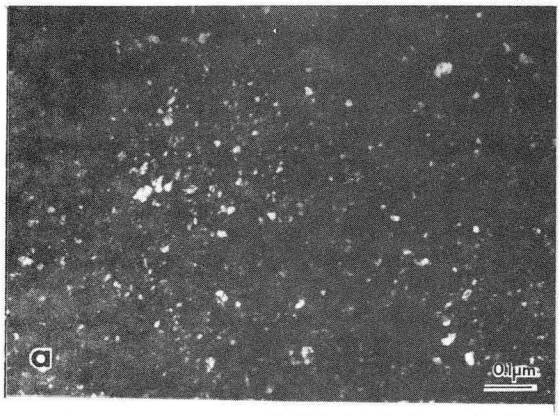


Figure 5. Dark field image of twins in [123] beam direction for : (a)M1 specimen 700°C/30 sec. RTA. (c)MH2, 700°C/30sec. + 1100°C/30sec . (d) FAS, furnace anneal simulation. (XBB-849-7284a)



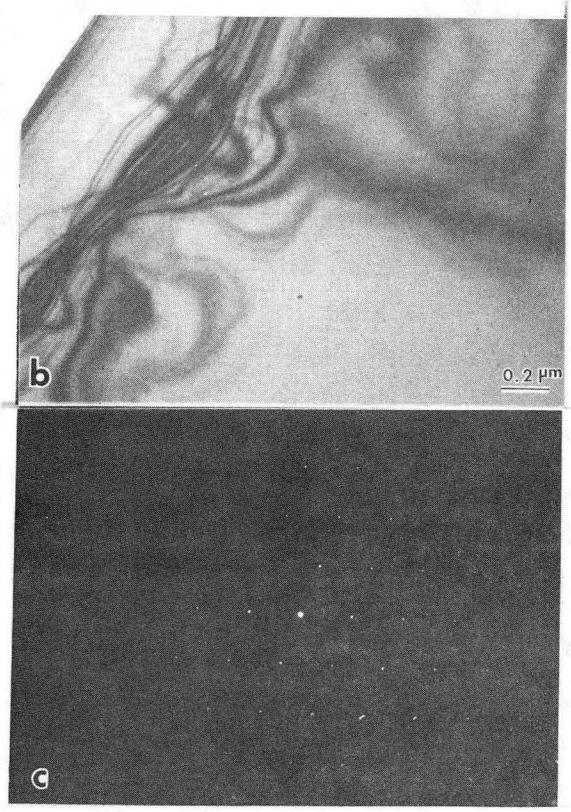


Figure 6. (a) Dark field image of twin in [123] beam direction for specimen furnace annealed at 550°C for 220 minutes. (b) Bright field image of defect free specimen in [110] beam direction,550°C/30min. + 1100°C/10min. (c) diffraction pattern of defect free specimen in [110] beam direction, shows no twin diffraction spots. (XBB 841-270a)

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