Self-Aligned Sources for Dislocation Nucleation: The Key to Low Threading Dislocation Densities in Compositionally Graded Thin Films Grown at Low Temperature

F. K. LeGoues
IBM Research Division, T. J. Watson Research Center, Yorktown Heights, New York 10598

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We show that, contrary to previous modeling and experiments, threading dislocation annihilation plays the dominant role in obtaining low threading dislocation densities during low temperature growth of compositionally graded SiGe layers. This is demonstrated by measuring the number of dislocations nucleated, which together with the density of threading dislocations, provides a direct measure of the number of annihilation events. Thread annihilation happens on a much larger scale than expected because the dislocation reproduction mechanism results in the "self-alignment" of the sources, leading to an (almost) perfect network of zigzagging dislocations.

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The possibility of obtaining new or better electronic properties for Si based materials through heteroepitaxy has led to considerable work concerning the formation of dislocations at strained interfaces, particularly the SiGe/Si interface [1–11]. Of critical importance for technological applications is the threading dislocation density (TDD) remaining in the film at the end of the growth. Recently, several techniques have been proposed that result in low TDD [1–4]. Particularly promising because of the enhanced electron mobilities observed in these structures [5,6] are results obtained with compositionally graded layers [1,2] grown under different conditions. In one case, shallow grading (about 10% Ge/μm) and elevated growth temperatures (950 °C) were used [1], while in the other case steep compositional grading (about 25% Ge per 0.5 μm) and low temperature (500 °C) were used [2]. Grading makes dislocation glide easier by allowing threading segments to overcome pinning points. Glide is also enhanced in the high temperature case compared to the low temperature one. On the other hand, in the low temperature case, nucleation of the dislocations is the controlling step [7], again rendering glide easier.

Improving glide improves the quality of the relaxed layer by allowing each dislocation to relieve the misfit over a longer distance, reducing the need to nucleate new dislocations. It also makes thread annihilation more likely. However, we show here that this is not sufficient to explain the TDD's of 10^4/cm^2 to 10^6/cm^2 obtained experimentally. In this Letter, we demonstrate that for the low temperature case, contrary to expectations [2,8], threading dislocation annihilation, and not enhanced glide, plays the determining role in obtaining low TDD. We show that the reproduction mechanism observed in these samples [the modified Frank-Read mechanism [2] (MFR)] leaves a trace of each nucleation event, directly observable by transmission electron microscopy (TEM). The number of annihilation events is thus measured by counting the number of threads nucleated, and subtracting the number of threads left at the end. The large scale annihilation measured in this manner is explained by the "self-alignment" of the nucleation sources, whose geometry forces threading segments moving on intersecting planes to meet and annihilate. An almost perfect zigzagging network of dislocations results. We show experimental evidence of this phenomenon.

Consider the case of Fig. 1(a). Three dislocations have nucleated in different parts of the sample. If the distance between dislocations *A* and *B* is less than or equal to the spacing needed to relieve the misfit, the driving force on *C*'s threading arm becomes zero when *C* moves into the vicinity of *A* and *B*, and the three dislocations pin each other. If *C* and *A* are gliding on the same plane, their threading arms annihilate [Fig. 1(b)], but Hull *et al.* [8] showed that, for random nucleation, this is statistically unlikely unless the TDD is higher than 10^8/cm^2. These statistics improve with higher temperature and slower growth [8], which may explain the high temperature, shallow gradient results. But the low temperature, steep grading case remains puzzling. In fact, the observation of TDD as low as 10^4/cm^2 had initially led to the conclusion that most of the threads had traveled the entire length of the wafer and "fallen off the edges" [2]. But this too is unlikely since it requires the nucleation of exactly the right number of dislocations and also that one end of the wafer "know" what the other end is doing, so as to avoid the situation depicted in Fig. 1(a).

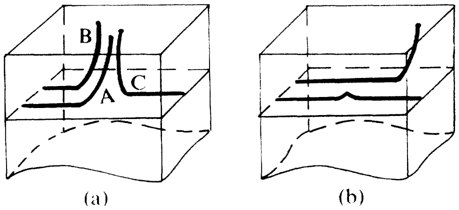

FIG. 1. Schematic representation of three randomly nucleated dislocations. (a) Dislocations *A*, *B*, and *C* glide on three different planes and have pinned each other. (b) Dislocations *A* and *C* glide on the same plane and their threading segments have annihilated.

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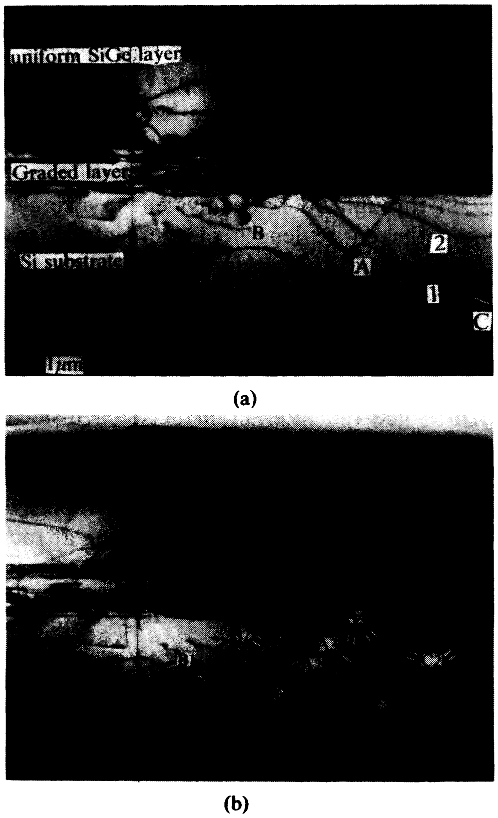
In order to determine whether thread annihilation or dislocations "falling off the edge" dominate, we need to measure the number of threads formed during relaxation, which is twice the number of dislocations nucleated. The number of annihilation events is the difference between this number and the number of threads left plus the number of dislocations needed to account for the relaxation. Unfortunately, the "post-mortem" type of experiments such as TEM, electron beam induced current, chemical etching, etc., only give a picture of the final microstructure, where, in most cases, the nucleation event is long lost in the tangle of dislocations present at the end.

Figure 2(a) shows a typical TEM cross-section micrograph of a compositionally graded sample grown at 500 °C [2]. The top layer contains 30% Ge, and was shown by x-ray diffraction [9] to be 90% relaxed, corresponding to a misfit between substrate and top layer of 1%. Two pileups (*A* and *B*) are in the Si substrate, typical of relaxation by the MFR mechanism. The MFR source corresponds to the intersection between two dislocations having the same Burgers vector and gliding on two different {111} planes [2]. From this intersection "corner dislocations" are generated, with threading arms moving in perpendicular directions [Fig. 3(a)]. In Fig. 2(b), both perpendicular sides of the corner pileups are visible (arrows *A*1 through *A*4 and *B*1 through *B*2), demonstrating that the cross section cuts through the two sources. Thus, the microstructure shown in Fig. 2(a) is the trace of two nucleation sources. The number of dislocations formed at each source is the number of segments extending in the thin direction of the cross section (the [110] direction in Fig. 2(b)), since the thin TEM sample may not retain all of the segments extending in the perpendicular direction. Consequently, for this mechanism, the final microstructure retains the trace of each nucleation event. The number of dislocations nucleated is the number of sources multiplied by the average number of dislocations formed at each source and is measured from the TEM cross-sectional micrograph in the manner described above. (Note that cross-sectional TEM is a poor way to obtain reliable statistics, since it probes such



(a)

(b)

FIG. 2. (a) Cross-sectional view with the electron beam exactly parallel to [110], showing the typical configuration of the MFR source. *A* and *B* show two pileups. *C* points to a third source, out of this field of view, which has injected dislocations 1 and 2. (b) Same area tilted about 30° around the [1$\bar{1}$0] direction, showing the segments of the corner dislocations that lie in the [110] direction. *A*1 through *A*4 show both sides of the corner dislocations in pileup *A*. *B*1 and *B*2 show the same thing for pileup *B*. *C*1 and *C*2 show that dislocations coming from *C* have zigzagged at *A*.

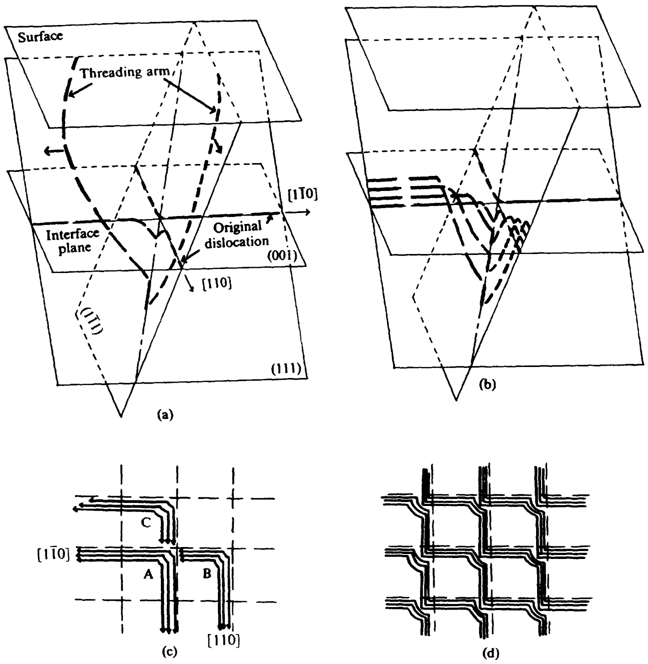


FIG. 3. Schematic representation of an MFR source, corresponding to the crossing of two original dislocations ($b = 1/2[10\bar{1}]$ in this case). (a) One half loop has formed with threading segments moving in perpendicular directions. (b) Same source, after formation of three dislocations, and the threading segments have moved away, showing the pileup in the substrate, similar to what is shown experimentally in Figs. 2 and 4. (c) Planar representation of the original network of dislocations, in dashed lines. Dislocations formed by the MFR mechanism at *A*, *B*, and *C* are represented as solid lines. See text for description. (d) Planar representation of the resulting zigzagging network of dislocations.

a small area of the sample, but we will see that the order of magnitude obtained in this manner is sufficient for our analysis.)

We find one source per square micron, with each source producing about 10 dislocations. Thus, for a 5 in. wafer, over 10^{11} dislocations have formed. This is orders of magnitude more dislocations than are needed to relax 1% misfit, which corresponds to a dislocation spacing of 200 Å, in two perpendicular directions. If every dislocation travels the entire length of the wafer, only 10^7 dislocations are needed. Consequently, the majority of dislocations have *not* moved to the edges. On average, the TDD is $10^6/\text{cm}^2$, or 10^8 threads per wafer (this is obtained by counting the number of threading dislocations in the TEM micrograph of the planar section of the samples). This demonstrates that a minimum of 99.9% of the threading dislocations have annihilated with each other. More importantly, this occurs when the TDD is several orders of magnitude lower than $10^8/\text{cm}^2$, the level below which Hull *et al.* [8] showed that annihilation was statistically unlikely.

Recently, Shiryaev [10] proposed that, if dislocations are generated through a simple Frank-Read mechanism, the probability of dislocations annihilation is 1 at infinite time. The case of the MFR mechanism is more complicated; each of the four possible Burgers vectors forms a specific corner configuration [Figs. 3(a) and 3(b)] where, along each of the two axes, all of the threads move in the same direction, and cannot ever annihilate. In order for annihilation to occur, two perpendicular segments have to meet at the intersection of their respective glide planes [segments coming from *B* and *C* meeting at *A* in Fig. 3(c)]. Shiryaev speculated that this was unlikely, since the two segments have to be at *A* simultaneously, or have to wait for each other. Yet, the experimental data show that annihilation not only occurs but it dominates the relaxation process.

In order to understand this phenomenon, we need to look further at the MFR source. Figure 3(c) shows one network of original dislocations having all the same Burgers vectors. Since there are four possible Burgers vectors of the $1/2\langle 110\rangle$ type, there are four such networks superimposed over each other. We will deal with this complication later. Let us assume that *A* is the first source to generate dislocations. Each thread travels a large distance, because there are few dislocations to pin it, and no strain has been relieved yet. When *B* starts operating, the threads moving in the [110] direction will travel long distances, similarly to those dislocations formed at *A*. On the other hand, the threading arms moving along the $[1\bar{1}0]$ direction soon meet an insurmountable obstacle in the pileup generated earlier at *A*. Indeed, dislocations of equal Burgers vectors repel each other, and the dislocations moving from *B* are meeting an entire pileup of dislocations of equal Burgers vectors. Further, in order to move past *A*, the dislocations nucleated at *B* have to extend in an area that is already substantially relaxed by the dislocations that have been previously formed. On the other hand, these dislocations can cross slip toward *C*, since this region is as yet unrelaxed. Eventually, *C* starts operating. Each dislocation moving from *C* in the $[1\bar{1}0]$ direction encounters a matching thread, coming from *B*, which is either " waiting" at *A*, or has cross slipped and is moving toward *C*. The two threads are attractive because they have antiparallel Burgers vectors. In the limit, the perfect network schematically represented in Fig. 3(d) results, where dislocations zigzag from one end of the wafer to the other, and all of their threading arms cancel out. This annihilation does not need to happen between neighboring sources, and all the sources need not have generated equal numbers of dislocations, since all the sources will be aligned, over long distances, on the original network of dislocations.

The resulting zigzagging network of dislocations is observed in cross-section samples. In Fig. 2(b), *C*1 and *C*2 show two dislocations, coming from source *C*, which have "zigzagged" away from pileup *A*. Figure 4 shows the same phenomenon, occurring on four aligned sources: Each dislocation encountering a previously formed pileup forms a new corner by annihilation, resulting in a network equivalent to one formed by one single source instead of the aligned array of sources. For example, dislocations *A*1 through *D*1 are equivalent to a single dislocation going the entire length of the picture, so that no threading segment is left at any of the pileups.

The presence of four intersecting networks of sources complicates the description of this process, but without altering the conclusion. Consider the four possible Burgers vectors, $1/2[101]$, $1/2[01\bar{1}]$, $1/2[011]$, and $1/2[10\bar{1}]$. Since the interaction between dislocations b_1 and b_2 varies as the cross product $b_1 b_2$, for each Burgers vector, the interaction with one of the other three is zero in one case, attractive in another, and repulsive in the third. If the interaction is either zero or attractive, the intersecting networks will have little effect on each other. If the interaction is repulsive, a pileup of one network

FIG. 4. Cross-sectional TEM showing a series of four aligned sources (see text). *A*, *B*, *C*, and *D* show four sources, similar to that seen in Fig. 3(b). *A*1, *B*1, *C*1, and *D*1 show dislocations nucleated at each of the sources.

may pin dislocations formed on the other network, but the pinning is weaker than in the case shown in Fig. 3(c) because the interaction is only half that between two dislocations of equal Burgers vectors and it does not happen at a corner, so that the dislocations will not extend in areas that are already relaxed. Thus, the four subnetworks of dislocations should multiply more or less independently of each other.

A more fundamental problem is that the original network of dislocations probably does not extend across the entire wafer. The annihilation can only happen in each area of "self-aligned" sources, resulting in regions of systematic pinning at the intersection of the different original networks, leading to rows of pinned threading dislocations. This may well be what is often observed as rows of pits in planar views [9], or as threading dislocation bunching in cross section which has also been observed by other groups [11].

We have shown that, because of its geometry, the MFR source leaves a trace of each nucleation event, which can be observed at the end of the relaxation process, even after considerable dislocation interaction has taken place. We thus count the number of nucleation events and measure the number of annihilation events. We show that during relaxation by the MFR mechanism, at least 99.9% of all of the threading segments formed are annihilated. This is due to the "self-alignment" of the sources, which provides each thread with a matching thread to annihilate. This may well be the easiest way to obtain low TDD since it does not rely on dislocations gliding for long distances and avoids pinning resulting from random nucleation. Also, unlike recently proposed ways to control the TDD it does not requires any additional processing step [3,4]. Finally, now that the self-alignment mechanism is understood, it may be possible to considerably improve the relaxed layer by understanding and controlling the formation of the initial dislocation network.

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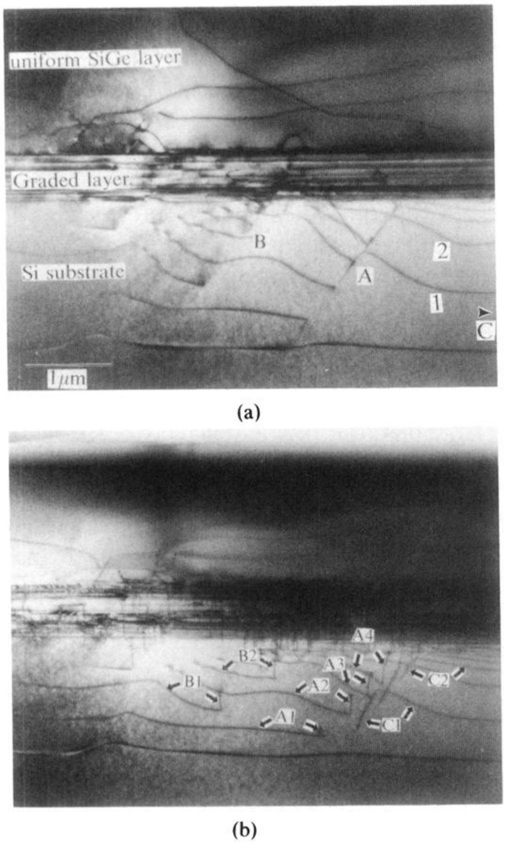

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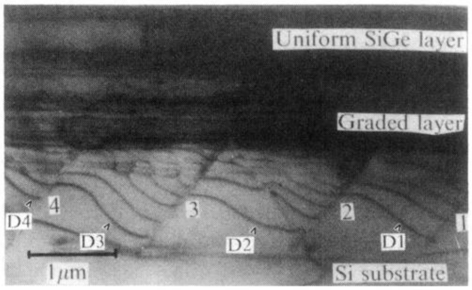

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