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SAND2000-0754

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Printed March 2000

Compliant Substrate Technology for Dissimilar Epitaxy

Jerrold A. Floro, Stephen R. Lee, Favid M. Follstaedt, and John F. Klem

Prepared by
Sandia National Laboratories
Albuquerque, New Mexico 87185 and Livermore, California 94550

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ABSTRACT

Many technologically interesting combinations of epitaxial films and substrates are unable to be exploited because of incompatibilities in the lattice parameter, thermal expansion rates, or chemical interactions. One approach to circumvent these problems that has received widespread attention is the compliant substrate. We review the concepts of the compliant substrate, the experimental signatures expected if compliancy behavior is occurring, and the facts and fictions associated with compliant substrate behavior. Our own experiments reveal no clear compliancy for heteroepitaxial growth of SiGe alloys on Si/Si twist-bonded substrates provided by Cornell University. These experiments have been limited, however, by a severe lack of substrates. We also review the worldwide progress on compliant substrates over the last three years. Definitive demonstrations of compliancy have yet to emerge, with one possible exception. *This is the final*

report for LDRD 3506630000, "Compliant Substrates for Epitaxial Intergration of Dissimilar Materials".

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Introduction

Strained-layer semiconductor films offer tremendous potential with regards to optoelectronic applications for high speed communications, mobile communications, sensing, and novel logic devices. It is an unfortunate reality that many of the possible film/substrate combinations that could be exploited technologically are “off limits” because of large differences in lattice parameters, chemical compatibilities, or thermal expansion rates. These mechanical, chemical, and thermal incompatibilities manifest themselves primarily in terms of lattice defects such as dislocations and antiphase boundaries, and in some cases through enhanced surface roughness. An additional limitation, from a production point of view, is money. Device manufacturers as a rule want the cheapest substrate possible. Freeing the heteroepitaxial world of the bonds of (near) lattice matching would vastly expand the types of *working* devices that could be grown. As a result, a great deal of effort has been expended finding schemes to integrate dissimilar film/substrate materials while preserving the perfection of the film layer. One such scheme receiving significant attention lately is the so-called *compliant substrate* approach.

Background

The compliant substrate approach was first proposed by Lo in 1991 [1]. The basic idea is to create a substrate that elastically complies, effectively absorbing the lattice mismatch strain of the epilayer film into itself. Figure 1 sketches the basic idea. In the common situation of a thin film on a much thicker substrate, essentially all the strain resides in the film and the substrate is unstrained, except for the small curvature imposed by the film. The strain partitioning in the film and substrate is given by [2]

$$\begin{aligned}\epsilon_f &= \frac{h_s}{h_s + h_f} \epsilon_m \\ \epsilon_s &= -\frac{h_f}{h_s + h_f} \epsilon_m\end{aligned}\tag{1}$$

where $h_{f(s)}$ is the film (substrate) thickness, $\epsilon_{f(s)}$ is the film (substrate) strain, ϵ_m is the mismatch strain, and the elastic constants of the film and substrate have been assumed identical. Since the substrate is typically of order 1000x thicker than the film, very little strain can reside in the substrate. However, equation (1) suggests that if the substrate thickness can be made to be the same order as the film thickness, then a significant fraction of the strain would be elastically shared by the substrate. In essence, the roles of the film and substrate are simply reversed. The

result would be a film having reduced strain and no additional defects, at the expense of a strained substrate, possibly containing additional defects (which may be permissible).

Figure 1. Attachment of a misfitting film to a substrate. *Left panel:* If the substrate is much thicker than the film, all deformation resides in the film. *Right panel:* If the film and substrate thicknesses are similar, both the film and substrate will deform, in opposite directions.

Of course, reality always intrudes in unpleasant ways. First, such a free-standing structure would curl up into a scroll. Second, a six-inch diameter substrate of thickness of order 100-1000 Å would both be rather delicate and rather expensive. The trick, then, is to fabricate a thin substrate attached to a stable, thicker substrate (a “handle wafer”) such that the thin section is

easily able to slide laterally relative to the thick part, thereby *elastically* accommodating strain during epilayer growth (perhaps followed by plasticity at a later stage), but at the same time suppressing curling or buckling of the epilayer/substrate layer. This would represent the ideal compliant substrate.

There were two “classic” experiments reported in the literature that were designed to produce compliant substrates, and the results of these experiments were sufficiently interesting to engender widespread excitement in the epitaxial growth community, including at Sandia, which eventually led to the establishment of this LDRD.

THE IBM APPROACH: In the first, sketched in Figure 2a, a strained SiGe alloy was deposited on a silicon-on-insulator (SOI) substrate, created by an oxygen implantation/annealing (“SIMOX”) process, and then thinned to produce a silicon overlayer less than 1000 Å thick [3,4]. After SiGe MBE growth, the films were annealed at temperatures above 700°C, and then characterized. X-ray diffraction indicated that significant strain relaxation occurred during annealing. Cross-section TEM (XTEM) revealed the presence of misfit dislocations at the SiGe/Si interface, as usual, but the key result was that the threading segments all appeared to pass *down* into the Si rather than up through the SiGe. The inference drawn was that the Si had become strained (via a compliant substrate process) and then sheared. However, it was never determined what mechanism would have *first* allowed the Si to move laterally. It was widely assumed that viscous flow of the oxide occurred, although it is highly unlikely that a SIMOX oxide, which is formed at temperatures above 1200°C, would flow at such low temperatures. The original investigators actually acknowledged this, instead suggesting that some sort of slip at the Si/SiO₂ interface occurred. Again, this seems rather unlikely, but cannot yet be discounted.

THE CORNELL APPROACH: A second experiment using a different type of substrate raised the excitement levels to even greater heights [5-7]. The compliant substrate in this case was essentially just two GaAs wafers bonded together with an in-plane twist misorientation, and then thinned from one side to create a thin single crystal layer joined to a handle wafer by a twist grain boundary (see Figure 2b). This structure was optimistically dubbed the “Compliant Universal Substrate”. While the concept was devised and carried out by Lo et al. at Cornell, Sandia played a role in this experiment by growing the epitaxial etch stop and bonding layers that allowed the thinning process to be carried out controllably. Subsequent epitaxial growth of on the GaAs twist-bonded substrates yielded epilayers that had much lower threading defect densities (as seen by XTEM) than in similar layers grown on monolithic GaAs. A similar result was obtained by Lo et al. for Ge on a Si-Si twist-bonded substrate [8]. In both cases a compliancy mechanism was invoked, wherein it was postulated that the twist-bonded interface had extensive regions where bonding was poor, permitting easier lateral slip of the twist-bonded layer. This actually has some precedent in grain boundary sliding mechanisms observed in stressed metal polycrystals. However, see the discussion that immediately follows.

Figure 2. Two approaches for compliant substrates. (a) Edge-on view of SOI-based compliant substrate structure used by IBM for growth of strained SiGe epilayers. (b) Plan-view of twist-boundary approach used by Cornell.

Both the IBM and Cornell reports, and much of the subsequent discussion in the growth community, were riddled with misconceptions about the elementary mechanics associated with compliant substrates. We now list the basic fictions, and corresponding facts, common to this field:

FICTION: Since the epilayer is under a state of biaxial stress due to lattice mismatch, the “compliant” layer must therefore be under stress, or strain, of the opposite sign, with magnitude given by equation (1), *and with h_s = the thickness of the compliant layer*. This implies that the compliant layer is “automatically” subject to stresses of the same order as the epilayer.

FACT: For any “compliant” layer mechanically bonded to a handle wafer, the magnitude of the strain is given by equation (1), *but with h_s = the thickness of the handle wafer*. Therefore the biaxial stresses in the compliant layer are typically three orders of magnitude smaller than in the epilayer. *The compliant layer must first slip or flow relative to the handle wafer in order to experience significant stresses.*

FICTION: There are large shear stresses present all across the epilayer/compliant layer interface, and across the compliant layer/handle wafer interface, that drive the aforementioned slip, or viscous flow, to occur.

FACT: Shear stresses are only present at the edges of the wafer. Elsewhere, the stress at the interfaces is purely biaxial (see the previous FACT for the magnitude of the biaxial stresses and strains at the various interfaces), i.e., the resolved shear components are identically zero. Therefore, *slip or flow can only start at the edges of the wafer, and then must propagate radially inward across the macroscopic dimensions of the sample.*

FICTION: Growth on a nominally compliant substrate structure that shows reduced defect density demonstrates that compliancy occurred.

FACT: Reduced defect density, while desirable, in no way serves to demonstrate substrate compliancy as envisioned in Figure 1. There are, in fact, three clear “signatures”, *all of which must be observed*, in order to definitively demonstrate that true substrate compliancy occurred:

1. **The epilayer must have reduced strain.**
2. **The lateral dimension of the epilayer/compliant layer must change relative to that of the underlying handle wafer.**
3. **The thin compliant substrate must then exhibit a state of *elastic* strain having opposite sign to the epilayer, and magnitude given by equation (1), with h_s = the thickness of the compliant layer. Eventually, the compliant substrate might undergo plastic deformation.**

The latter two signatures serve to demonstrate the same thing: that lateral slip or flow occurred in the compliant substrate. To our knowledge, signature (2) has never been observed (at least the elastic part), and there is perhaps only one observation of signature (3) — from an experiment at the Naval Research Labs — that we shall discuss near the end of this report. It is an unfortunate fact that few in the compliant substrate community have recognized the importance of these signatures in defining whether compliancy has occurred.

Based on the interesting results of the IBM and Cornell experiments, an LDRD was proposed whose goal was to directly evaluate the mechanisms for relaxation in twist-bonded substrates, and to exploit growth on these substrates for mission-critical device fabrication. Cornell was to collaborate with us by providing twist bonded GaAs and Si substrates for growth and evaluation by Sandia. Unfortunately, the lack of substrates proved to be a show stopper.

Project Organization/ Accomplishments

In this LDRD our primary purpose was to first establish sound scientific proof of compliant substrate behavior, and then to determine the mechanism for compliancy. This was to be

followed by fabrication of novel III-V-based devices on compliant substrates that could not be *successfully* fabricated on a standard monolithic substrate. The compliant substrates were to be twist-bonded Si and GaAs provided by Prof. Y.-H. Lo at Cornell University.

The basic scientific approach was to grow identical strain-layer films on both compliant and monolithic substrates while monitoring film stress in situ, and in real-time, using our Multi-beam Optical Stress Sensor (MOSS). It was expected that compliant behavior would exhibit significantly different relaxation kinetics than would be observed for films on monolithic substrates. Growths of $\text{Si}_{1-x}\text{Ge}_x$ on Si and InAsSb on GaAs were planned. After growth, careful analysis of the final strain states of both the film and substrate, using x-ray reciprocal space mapping (known as KMAP), and characterization of the final microstructure using cross-section transmission electron microscopy (XTEM), were to be carried out. Experimental matrices varying strain (through alloy composition) and kinetics (through growth temperature) were designed. A series of experiments examining strain relaxation as a function of the thickness of the twist-bonded overlayers was also desired.

Results

Precharacterization

We first wanted to characterize as-received twist-bonded substrates from Cornell. As these substrates were clearly quite difficult to produce, we chose KMAP as a means to non-destructively detect and characterize the twist-bonded layer. Two different (001) Si-on-Si twist-bonded samples were examined by x-ray diffraction. The first (designated sample # CUSi09) was intended to be a 180 Å-thick Si thin film bonded to a bulk-Si handle wafer at a ~30 degree twist angle. The second (designated sample # CUSi15) was intended to be a 40-angstrom-thick Si thin film bonded to a bulk-Si handle wafer at a ~45-degree twist angle. The twist angle between the thin bonded film and the handle wafer spatially separates all of the normally overlapping asymmetric-diffraction reflections of the handle wafer and the bonded film. The separated asymmetric reflections [(113) or (224)] of the bonded film were used to detect the presence of the bonded layer and evaluate its structure without interference due to diffraction from the substrate. These two twist-bonded-Si samples were examined as-received from Cornell University.

The (113) reflection/[1-10] zone-axis reciprocal-space maps of CUSi09 and CUSi15 appear in Figures 3a and 3b, respectively. Diffraction from the thinned Si layer of CUSi09 was readily detected indicating successful fabrication of the twist-bonded Si layer; Figure 3a is the result of a 56-hour-long diffuse scattering scan that maps the diffracted-intensity fine structure about the (113) reflection of the thinned crystal. Modulated thickness fringes observed along the truncation rod in Figure 3a indicate an actual thickness for the bonded Si layer of 220 Å in reasonable agreement with the target-value of 180 Å. The full-width-half-maximum of the truncation rod as measured near the center of the pattern yields a nominal lateral coherence

Figure 3. X-ray reciprocal space maps of twist-bonded substrates. The maps are (113) reflection/[1-10] zone-axis from (a) CUSi09 and (b) CUSi15. The twist-bonded overlayer is clearly observed in CUSi09, but is absent in CUSi15.

length for the bonded, thinned crystal of 1850 Å. This result suggests that either laterally inhomogeneous strain fields, or laterally varying crystal misorientation, exists in the bonded film on a spatial scale comparable to the coherence length. A broader diffuse pattern (with the broadening occurring transverse to the scattering vector) is also seen in Figure 3a adjacent to the main Bragg peak. This last component of the pattern in Figure 3a may arise due to x-rays scattering from the oscillating displacement fields associated with the screw dislocations that form the twist boundary.

In contrast to CUSi09, diffraction from the thinned 40-angstrom Si layer of CUSi15 could not be detected, as seen in Figure 3b. Kinematic diffraction theory estimates of the change in

intensity due to the change in layer thickness for CUSi15 relative to CUSi09 along with consideration of the signal to noise ratio seen for Figure 3a) suggests that a weak signal should have been observed if the 40-angstrom layer were indeed present. Either the bonded film is over-thinned (eliminating the entire bonded layer) or perhaps the nominal orientation of the crystal is incorrect. Thickness control problems during the etching and polishing steps that were used to thin the twist-bonded wafer-couple to the final film thickness were observed over the entire course of this study. It is therefore quite plausible that for CUSi15 the entire twist-bonded layer was unintentionally removed by polishing during twist-bonded substrate preparation at Cornell.

This work demonstrated that KMAP can resolve the thickness, orientation, and strain distribution for twist overlayers less than 100 Å thick.

SiGe Growth

Si_{1-x}Ge_x alloy growths were carried out on two Si/Si samples provided by Cornell, CUSi09, described above, and on an earlier sample having a nominal 150 Å overlayer bonded with 45° twist rotation (this sample was not precharacterized by KMAP). For both these samples, a standard ex situ wet chemical clean (the “modified IMEC/Shiraki clean”) was employed. We first determined that this cleaning procedure removed a maximum of 50 Å of material. This was done by growing a Si layer on a Ge monolayer marker on Si (001). The thickness of one portion of the film was measured as-grown using x-ray reflectivity, while the other half was first subjected to the cleaning procedure and then similarly measured.

Reflection high energy electron diffraction (RHEED) from these films after in situ oxide desorption was performed on the twist-bonded overlayer and on the exposed regions of the handle wafer. The pattern was clearly observed to rotate to a different azimuth in the two regions, indicating that the twist overlayers survived the cleaning process (see Figure 4).

Figure 4. RHEED patterns from a CU substrate. The nominal overlayer thickness was 150 Å, but actual thickness was later found to be 650 Å. (a) Pattern from handle wafer region, corresponds to a <110> azimuth. (b) Pattern from twist-bonded overlayer region, corresponds to a <100> azimuth.

Real-time wafer curvature data is shown in Figure 5 for growth and annealing of $\text{Si}_{1.7}\text{Ge}_3$ on the nominal 150 Å Si/Si wafer, and on a monolithic Si wafer. The growth rate was 1 Å/s and the growth temperature was 550°C, which favors 2D planar growth followed by dislocation formation over 3D island growth at this level of strain. As is observed in the figure, the relaxation kinetics are essentially identical in both films, inconsistent with any compliancy mechanism in the Si/Si sample. It was learned later that the twist overlayer was actually about 650 Å thick. Nonetheless, some elastic deformation of the twist layer was expected, e.g., at 650 Å film thickness we would expect about half the film strain to be taken up by the compliant substrate. **No such compliancy behavior was observed.**

Figure 5. Real-time curvature data. Curvature, which is proportional to the stress*thickness product of the film, was measured *in situ* during MBE growth and annealing of $\text{Si}_{0.7}\text{Ge}_{0.3}$ alloys on bulk Si (001) and a Si (001) twist-bonded (CU) substrate.

Growth of $\text{Si}_{0.4}\text{Ge}_{0.6}$ on CUSi09 was performed at 1 Å/s, 600°C, a regime that promotes island formation, on both Si/Si and monolithic Si. Unfortunately, useful MOSS data was not obtained from the growth on Si/Si. RHEED from both films indicated essentially identical surface morphological evolution. Post-deposition KMAP revealed that the film on Si/Si was 75% relaxed, while the film on monolithic Si was 90% relaxed. XTEM of the film on Si/Si

revealed a complex, highly defective microstructure with two domains of different orientation interwoven through the film (see Figure 6). This structure suggests that the Si twist layer was discontinuous, so that many regions of the handle wafer were exposed through “pinholes” for epitaxial growth. Since this did not show up in RHEED, it is presumed that the pinholes had a steep enough aspect ratio that the glancing incidence RHEED could not illuminate the handle wafer.

Figure 6. XTEM micrographs of $\text{Si}_{0.6}\text{Ge}_{0.4}$ domains that grew in two different orientations. The domains were rotated by 21° , corresponding to growth on the twist-layer and growth on the handle wafer. Substrate CUSi09.

Some aspects of this film were intriguing. In particular, it appeared in some micrographs that the domains grown on the twist layer contained fewer threading defects than the domains on the handle wafer (see Figure 7). In cross-section, however, this determination was rather difficult to make quantitative. This was certainly a rather unusual example of the desired outcome, e.g., reduced defect density, but was not construed to be a definitive demonstration of a compliancy mechanism. A possible explanation will be presented later.

RHEED from CUSi15 confirmed that no twist layer was present, in agreement with the KMAP results. A modified cleaning procedure was employed in this case to ensure that if a layer of thickness greater than about $20 \text{ \AA}$ were present, it would not be removed during cleaning.

In summary, no clear demonstration of any compliancy effect was observed during growth of SiGe strained layers on $650 \text{ \AA}$ thick and $220 \text{ \AA}$ thick Si/Si twist-bonded substrates. In the latter sample, a possible reduction in threading defect density was noted in a bicrystalline structure formed due to pinholes in the twist layer.

InAsSb Growth

Several III-V growths for this project were carried out on GaAs/GaAs twist-bonded substrates from Cornell. Unfortunately, RHEED and post-deposition analysis confirmed that no twist overlayer was present on any of these samples. A commercial MOSS system was

purchased, installed, and aligned on the EPI chamber in the CSRL, but was not used for

Figure 7. XTEM micrograph of Si_{0.6}Ge_{0.4} domain aligned with twist-bonded layer. The protruding domain seems to have lower threading defect density than the domains aligned with the handle wafer. Substrate CUSi09.

compliance measurements since no successful GaAs/GaAs substrates were ever obtained from Cornell.

Bonding at Sandia

A separate LDRD was already in place at Sandia to examine wafer bonding processes. It was hoped that we might collaborate with this project to produce our own twist-bonded substrates, since the Cornell supply was unreliable. However, difficulties encountered in the other LDRD did not make this collaboration possible. Furthermore, results from groups at University of Wisconsin and Max-Planck indicated that making these twist-bonded substrates required a very steep learning curve and a great deal of dedicated investment in time and equipment. Therefore it was decided that we would not further pursue our own bonding/fabrication effort.

Concurrent Results from Other Laboratories

Here we briefly, but critically, review the status of the field up through the end of calendar year 1999. Several groups have maintained active efforts attempting to fabricate and grow upon compliant substrate structures. The *Brown Group* has brought some theoretical sanity to the field through straightforward calculations of critical thicknesses and viscoplastic kinetics. The

Max-Planck Group, which specializes in wafer bonding, has brought a methodical approach to the field, examining the behavior of strained layers grown on twist-bonded substrates in great experimental and theoretical detail. They have not found any clear evidence of true compliancy behavior. The *Wisconsin Group* has focused on using low viscosity glasses as bonding layers, in order to obtain compliancy via glass flow as was envisioned (but not obtained) in the original IBM work [3,4]. So far, some interesting, albeit limited, results have been obtained that may be consistent with compliancy. The *Georgia Tech Group* has used low melting point metal bonding layers between GaAs substrates and handle wafers. While there has been an effect on epilayer relaxation kinetics, it is not at all clear that these effects are consistent with compliancy. The *Columbia Group* has recreated the IBM SiGe/SOI structure and demonstrated a significant reduction in defect density. The mechanism is not clear, but the apparent result is impressive. The *Cornell Group* which pioneered the twist-bonded structure, has largely ceased direct activities in this area, although some collaborative work has continued. The *UCSB Group* has obtained recent results that may suggest compliancy. The *NRL Group* has also recently obtained a fascinating result that is consistent with compliancy. More details on the results obtained by all these groups are given below.

The Brown Group (BG)

BG performed a detailed energy analysis to derive a critical thickness criterion applicable to epilayers on compliant substrates [2]. They showed that thin compliant substrates could indeed raise the critical thickness for introduction of misfit dislocations significantly by elastic strain partitioning from the epilayer into the substrate.

BG also carried out a 1D viscoelastic/kinetic analysis applicable to the IBM structure (or the WG structure described below) [9]. Even using generously high viscosities characteristic of BSG glasses, BG found that flow-based relaxation, which occurred radially inward across a 2 inch diameter wafer, required of order days to produce the observed degree of relaxation (rather than hours as in the IBM experiment).

The Max-Planck Group (MPG)

MPG has carried out a major effort in wafer bonding technology in the last decade [10]. As an offshoot of this effort, MPG evaluated the fabrication of twist-bonded substrates, particular GaAs/GaAs, and the subsequent growth, strain state, and microstructure of strained epilayers grown on the twist-bonded layers. Prior to discussing this, it is worth noting that MPG has provided several theoretical discussions of how twist-bonded substrates might relax epilayer strain, starting with the assumption that the classic compliant behavior is NOT occurring. They suggested three ideas: (1) screw dislocations that compose the twist-interface getter impurities that lead to the generation of defects in the epilayer [11]; (2) screw dislocations drive spatially

correlated generation of misfit dislocations, resulting in a decrease in threading defect density in the epilayer [11]; and (3) screw dislocations split to form 60° dislocation dipole networks that shear the twist-bonded layer, thus providing plastic compliancy of the substrate [12]. While the first two concepts were discussed only qualitatively, the latter idea was worked out in great detail. The mechanisms suggested appear reasonable, albeit somewhat complex to be working simultaneously over macroscopic distances. A more critical issue, however, is that the dislocation splitting processes suggested require that the twist-bonded layer be under significant stress in order to separate the dislocation dipole. The authors implicitly seem to suggest that the stress is equal to that expected in a free-standing bilayer (e.g., Figure 1), i.e., with the bonded layer having the opposite sign of stress in the epilayer, with magnitude given by equation (1). But this is circular reasoning, since the bonded must first slip relative to the handle wafer in order that such a stress exist. Prior to slip, only the very small stress associated with wafer curvature (given by equation (1), but with h_s = the thickness of the handle wafer) is present at the twist interface.

MPG performed two rather illuminating sets of experiments. In the first [13], InP layers were grown on 100 Å-GaAs/GaAs (14°) twist-bonded substrate (lattice mismatch strain = 3.5%). The thickness refers to the thickness of the GaAs twist-bonded layer on top of the handle wafer, while the angle refers to the twist angle. TEM revealed that the GaAs/GaAs substrate contained a high density of pinholes where the handle wafer was exposed. InP growth resulted in a bicrystalline film structure with domains having either the orientation of the twist layer or the handle, therefore, the domains were rotated by 14° with respect to one another. X-ray diffraction (XRD) revealed that both the domain variants were highly strain relaxed, while TEM revealed the presence of a misfit network in the InP/GaAs interface. Interestingly, the threading defect density within the domains was very low, which is, of course, the desired outcome for a “compliant” substrate. However, in this case, the low threading density did not arise from true substrate compliance, but was attributed to gettering of the threads at the grain boundaries separating the two rotational variants. *This result is very similar to what we observed with SiGe grown on a Si/Si twist-bonded wafers from Cornell.*

The second experiments [14] involved the growth of InGaAs (7% In) on 300 Å-GaAs/GaAs (6°). The twist-bonded substrate in this case was pinhole-free over large areas of substrate. InGaAs epilayers 400 Å and 3000 Å thick were grown. The 400 Å film remained pseudomorphic, while the 3000 Å film relaxed via misfit dislocations terminated by upward threading segments. Nothing in the dislocation configuration suggested compliancy had occurred. In the same paper, the kinetics of grain boundary sliding, as a paradigm for compliancy, were discussed. While the authors again incorrectly invoke the presence of a misfit stress in the twist-bonded layer, they rightly point out that the *shear* stress at the twist interface is negligible (they estimate a shear stress of 1 KPa, which is small, but a better estimate would be

only 1 Pa!). Thus a process related to grain boundary sliding is extremely unlikely to be occurring on realistic time scales.

In sum, MPG has found no evidence for relaxation via true compliancy.

The Wisconsin Group (WG)

WG's approach has been to fabricate semiconductor (mostly GaAs) on insulator substrates, where the insulator was a borosilicate glass with a high boron fraction, having viscosity's at least 6 orders of magnitude smaller than thermal oxide. The structures were formed by bond-and-etchback techniques [15]. WG also fabricated GaAs/GaAs twist-bonded substrates for comparison. InGaAs films (3% strain) were grown by OMVPE on bulk GaAs substrates, 100 Å-GaAs/BSG substrates, and 100 Å-GaAs/GaAs (12°) twist-bonded substrates. XRD analysis showed that the films grown on the glass-bonded substrates had the narrowest strain distribution (as measured by the full width at half max associated with the diffraction spectrum parallel to an 004 vector) [16]. The peak widths were 160, 280, and 540 arcsec for InGaAs on glass-bonded GaAs, bulk GaAs, and twist-bonded GaAs, respectively. Interestingly, mosaic widths were similar in all three cases. The degree of strain relaxation of the 3 μm thick epilayers unfortunately were not specified. Surface roughness was measured by atomic force microscopy (AFM), where it was found that the RMS roughnesses ranked as 9.5 nm, 42 nm, and 252 nm for InGaAs on glass-bonded GaAs, bulk GaAs, and twist-bonded GaAs, respectively. TEM identified the presence of a misfit dislocation network at the InGaAs/GaAs interface [17]. While they note that the density of dislocations was nearly an order of magnitude too small for a *fully* relaxed film, again they did not report measurements on the degree of relaxation. They also did not show TEM results for the films on bulk GaAs for intelligent comparison. *If* the film on the glass-bonded substrate was fully relaxed, which is certainly possible given the large thickness and In content (45%), then the TEM data does suggest that some other relaxation mechanism was operating.

In summary, WG have produced a possible result of interest, but sufficient information to be definitive is still lacking.

The Georgia Tech Group (GTG)

GTG created compliant substrates by bonding 3000 Å thick GaAs layers to GaAs handle wafers with an intervening In/Ga alloy layer that is (presumably) liquid during MBE growth of InGaAs (7% In) epilayers at the 520°C growth temperature used [18-21]. This might be expected to provide an ideal structure to test compliancy, since the liquid metal should not support shear, although the GaAs substrate layer is relatively thick at 3000 Å. This is a problem, since the Matthews-Blakeslee equilibrium critical thickness for the alloys grown here is only of

order 100 Å. Thus the epilayer should undergo standard dislocation introduction long before compliancy effects are significant, unless kinetic constraints keep the epilayer metastable. To check this, GTG also grew the same InGaAs alloys on bulk GaAs and found that significant relaxation via misfit dislocations occurred above 2000 Å film thickness. Therefore, for growth on the compliant substrates, at 2000 Å film thickness on a 3000 Å thick substrate, equation (1) predicts that about 40% of the mismatch strain in the epilayer should have been coupled into the substrate. However, GTG observed no strain relief at all on the compliant substrate at 2000 Å. Relaxation occurred only after 3000 Å film thickness! The subsequent relaxation kinetics were retarded as well for films grown on the compliant substrate vs. the bulk substrate. This is inconsistent with compliancy occurring in the liquid metal layer. GTG have not explained this, or even explicitly recognized the inconsistency [18]. GTG have also claimed to see the GaAs substrate layer in the opposite sign of stress, one of the “signatures” of compliancy [20]. Unfortunately, this claim showed up only in a footnote as “unpublished”.

The Colombia Group (ClG)

ClG essentially repeated the basic structure of the IBM group: they fabricated an SOI substrate with a 200 Å Si layer bonded to a thermal oxide acting as the compliant substrate [22]. On this substrate they grew 1 µm of Si₆Ge₄ (1.6 % mismatch strain) at 500°C. For control, a similar film was grown on bulk Si. Cross-section TEM indicated a reduction in threading dislocation density from 10¹¹ cm⁻² on bulk Si to 10⁶ cm⁻² on SOI, a reduction in defect density of five orders of magnitude! Unfortunately, only one very terse article, containing only a pair of XTEM images and no XRD, is all that has been published of this apparently spectacular result.

The Cornell Group (CnG)

CnG (Y. H. Lo) pioneered the original concept of the practical compliant substrate [1] and produced the first twist-bonded “compliant universal substrates” of GaAs/GaAs and Si/Si [5-7]. Initial results suggested major reductions in threading defect densities for strained layer growth on twist-bonded substrates vs. bulk substrates. As discussed at the beginning of this report, the initial collaboration between CnG and Sandia on the production of these samples eventually led to the formation of this LDRD, with CnG set to supply the compliant substrates. Unfortunately, CnG was unable to supply any reliable twist-bonded substrates (over 2 years we obtained five substrates, only one of which actually had a useful twist layer – the pinhole sample). At the current time (March, 2000), CnG apparently does not produce any further twist-bonded substrates, even though Lo has formed a startup company for wafer bonding.

The UCSB Group (UCSB)

UCSB has grown 5 monolayer (ML) thick InAs epilayers on GaAs (111)A substrates (mismatch strain = 7%), using different GaAs buffer layer thicknesses ranging from 1500 ML to only 10 ML [23]. Using in situ scanning tunneling microscopy (STM), they find a fully developed network of pure edge dislocations at the expected spacing for the misfit. The interesting observation is that the corrugation height associated with the misfits varies with the buffer layer thickness. They claim that this behavior is expected if the buffer layers behave compliantly, i.e., as if the bottom surface of the buffer layer is free. They support this claim with valence force field calculations of the structure, both as a function of buffer thickness and as a function of epilayer thickness. They also show experimental measurements of the corrugation amplitude for a range of epilayer thicknesses that agree well with the experiments.

How the GaAs buffer on a GaAs substrate can behave compliantly is unknown. UCSB suggests that either (1) the buffer was slightly misoriented with respect to the bulk wafer, thereby allowing it to act like a twist-bonded substrate, or (2) contamination at the buffer/substrate interface somehow affects the mechanical response. It is difficult to give much credence to item (1) in light of the evidence against compliancy effects in *deliberately* twist-bonded wafers and since the GaAs buffer is expected to be coherent and epitaxial with the substrate. Item (2) provides a convenient but unenlightening explanation. In either case, it is difficult to reconcile how the GaAs buffer /GaAs substrate interface can behave as if it were a free surface *in the vertical direction*, which is presumably required in order to explain the changes observed in the vertical corrugation amplitude at the InAs surface.

The NRL Group (NRLG)

Like Max-Planck, NRLG has carried out a robust effort in wafer-bonding. NRLG recently used bonding combined with the Smart-Cut process to create a $300 \text{ \AA} \text{Si}_{0.7}\text{Ge}_{0.3}/40 \text{ \AA} \text{Si}/\text{insulator-on-Si}$ structure [24]. The insulator was either thermal oxide or BPSG. As-bonded, the SiGe films retained their coherency with the substrate. For the film bonded to thermal oxide, a 975°C anneal results in 60% relaxation of the alloy. However, TEM revealed no misfit dislocations at the interface. The paper did not speculate as to how the film relaxed. Macroscopic flow of the oxide is excluded since no length change was observed in the overlayers relative to the handle wafer. Another explanation developed at Sandia is that the misfit cores are drawn to the Si/SiO₂ interface by image forces. At the crystal/amorphous interface, the cores can “pop out” of the epilayer just as they would at a free surface, if the oxide can flow *locally* to fill in the slip step that is created at the interface.

Annealing of the SiGe/Si films bonded to BPSG leads to extreme buckling of the film for anneals above the glass transition temperature of the BPSG, estimated to be 625°C for the composition used. The buckling took the form of an ordered sinusoid above 750°C . The XRD pattern vanished as the lattice planes tilted over 5° . The change in length of the film due to

buckling was determined to be 1.03% which relaxes 85% of the 1.2% misfit strain. Similar SiGe/Si/BPSG films were formed on patterned mesas. Here the film was able to expand laterally in a planar fashion off the edges of the mesa. XRD confirmed an 85% relaxation in this case.

This experiment may represent the clearest demonstration of the originally intended compliancy effect. What is still to be understood, however, is whether the kinetics of the buckling process make sense, since it would still be expected to proceed inwards from the edges of the wafer.

Summary of findings

Both at Sandia, and worldwide, significant effort has been expended over the last three years in pursuit of the compliancy "holy grail". Despite this, it seems that we are in much the same situation that we occupied in three years ago: there have been many results that tantalize, seeming to suggest compliant-like behavior, but falling frustratingly short of providing the definitive data that could prove the mechanism. Our own work has not shown any compliancy effect, although in one case a localized reduction in defect density in a pinhole structure was observed. However, this is not believed to result from the classic compliancy mechanism. Much of the work reported in the literature is still embedded in naïve, confusing, and uninformed discussion of compliancy mechanics and dynamics. **Nonetheless there is mounting evidence that, by imposing a disruption of the crystalline lattice of the substrate in a region near the epitaxial growth front, it is possible to strongly modify the overall process of strain relaxation. This is not likely the result of compliancy mechanisms as originally envisioned by Lo and others.** The actual mechanisms by which reductions in threading defect density occur are clearly more complex, and probably relate more to how dislocations nucleate, multiply, and respond to local image forces in non-monolithic structures.

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