Interfacial study of Si–Ge multilayers grown using ultrahigh-vacuum chemical vapor deposition

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Abstract

In this study, we used scanning probe microscopy to observe the abrasion damage of annealed Si–Ge superlattices. Specimens were more amenable to plastic deformation after annealing, thereby providing higher friction coefficients as a result of decreased strain energy. Restricted dislocation movement appeared not only in the combined Si–Ge superlattices but also at the interfaces, suggesting that thermal treatment could decrease the shear resistance. The component damage and oscillation events of the specimen that had been treated only at room temperature were more serious than those of the diffusion annealing specimens.

Keywords：Superlattices; Chemical vapor deposition; Friction; Diffusion annealing

1. Introduction

The epitaxial growth of thin films is an attractive process because the resulting materials possess unique physical characteristics. The operation of such superlattices is dependent upon the interaction of adjacent epitaxially grown layers featuring different electronic, optoelectronic, and thermal properties [1–3]. The presence of a large density of epitaxial interfaces in a device will severely degrade its thermoelectric cooling, power conversion, and heat transport. The development of strained-layer superlattices has led to their application in Si-based microdevices [4]. Si–Ge strained-layer superlattice films can be prepared using both reduced-pressure chemical vapor deposition (RPCVD) and ultrahigh-vacuum chemical vapor deposition (UHVCVD) in such a manner that the lattice mismatch of the two semiconductors can be accommodated. Si–Ge films have been characterized in terms of their Si–Ge interdiffusion [4–8]. In many cases, annealing treatment has the potential to reveal reliable quantitative information regarding the behavior of periodic multilayered Si–Ge structures. The mechanical characteristics of artificial multilayer films appear to correlate strongly with their geometrical dimensions [9].

Several reports suggest that epitaxial Si–Ge films can exhibit slightly enhanced mechanical properties after thermal treatment, as a result of misfit dislocation propagation, which can increase their hardness [10–12]. The mechanism from misfit dislocations to threading dislocations of crystal defects between the Si–Ge layer and the Si substrate can be determined through nanomechanical tests. There are, however, two key materials aspects limiting the thermal behavior that epitaxial films can endure during thermal processing: interdiffusion and strain relaxation. In a previous study [13], Si–Ge superlattices films were grown on GaAs substrates subsequent to superlattice growth; the Si or Ge layers introduced a large density of dislocations and stacking faults. The resulting plastically deformed Si–Ge material exhibited a thermal conductivity that approached the low values characteristic of amorphous solids. In this context, we would like to study the stress and deformation fields in the vicinity of the contact, but it is not a simple task because of the nanometer-scale periodicity of the Si and Ge layers. The periodical structures of annealed Si and Ge layers remain poorly understood in terms of their behavior during indentation processes. In this study, we used several cycles of UHVCVD growth to deposit a number of (nanometer-period) Si–Ge layers. We then investigated the effect of thermal treatment on the internal nanotribological behavior. Herein, we discuss the relationship between the quality and strain relaxation of the Si and Ge layers.

2.Experimental

In the deposition process, a Si–Ge superlattice structure were synthesized using the following procedure: (i) P-type Si(1 0 0) wafers were processed to standard Radio Corporation of American (RCA) cleaning and dipping in an HF:H2O (1:50) bath for 15 s. Afterward, specimens were conducted into the load-lock chamber of a UHVCVD system. (ii) A 5-nm-thick Si and Ge layer was synthesized at 665 °C for ca. 2 min from a mixture of pure SiH4 (85 sccm) and GeH4 (15 sccm) gases under a vacuum of 10–7 mbar. (iii) The Si–Ge superlattice specimen was completed by repeating steps (i) and (ii) five cycles; the total thickness was ca. 50 nm. (iv) The specimen (without annealing so-called room temperature (RT) condition) was subjected to ex-situ annealing treatment in a furnace under N2 gas for 30 min (at 400, 500, or 600 °C).

To identify the nanotribological properties of the specimens, a nanoscratch system was employed using atomic force microscopy (AFM, Digital Instruments Nanoscope III) in conjunction with a nanoindentation measurement system (Hysitron). A constant scan speed of 2 μm s–1 was applied. To determine the fracture and abrasion of the Si–Ge superlattices initiated at low-ramped-force modes of sliding cycles, samples were subjected to ramped loads of 2000 and 6000 μN (the start and end points from tip are marked by arrows). Using this approach, images corresponding to the surface profiles were obtained and 10-μm-long scratches were formed in the ramped-forces mode. After scratching, the wear tracks were imaged through ex-situ AFM examinations.

3. Results and discussion

Fig. 1 shows AFM images of a Si–Ge superlattice deposited on the Si substrate at (a) RT and thermal treatment at (b) 400, (c) 500, and (d) 600 °C. These specimens had been scratched at two different ramped forces (2000 and 6000 μN). We suggest that pure elastic contact dominated at the low ramped force (2000 μN), based on a slightly machined surface with cracks appearing at the end point. A surface morphology with elastic tracks at the end of the scratch functions was evident at a ramped force of 2000 μN. Henceforth, the annealed samples exhibited more pile-up near the middle of the sliding line (6000 μN) than did the specimen at RT. In all the cases, regions of pile-up are recorded in the tracking line. In a crystalline material, release of the scratching load is bound to reflect in the change in the lateral compressed volume. The reason for pile-up is the strong interdiffusion from the annealing treatment. Thus, we examined the role of the adhesion force on the nanomechanical damage and investigated the pile-up mechanism in greater detail.

Fig. 2 displays typical plots of the coefficient of friction (μ) with respect to the scratch duration (ramped force: 2000 or 6000 μN) for the specimens annealed at various temperatures; these samples were ramped over 10 μm at a constant scan rate of 2 μm s-1. For the specimens annealed at the various temperatures and subjected to a ramped force of 2000 μN, the friction traces were slightly increased at 25 s and exhibited a raised curve in each case. In addition, μ increased from the annealing treatment, particular for the condition of 600 °C (Fig. 2a). The increasing curve is ex-situ to the artificial deformation response shown in Fig. 1 during the lateral force load; the possible presence of the elastic/plastic contact translation state is noted.

For the corresponding specimens subjected to a ramped force of 6000 μN, the plots show period oscillations under the friction force during the 15 －38 s of the experiment in both the on- and off-load scans. However, the annealed specimens revealed more gradual increases in the friction traces than did the specimen at RT. The friction traces at a ramped load of 6000 μN exhibited more sudden oscillating fluctuations than did those obtained at 2000 μN. Such plots obtained using the nanoscratch technique [5, 8] often feature three main regions: settling, oscillation, and unwanted fluctuations. We conclude that the lower degrees of adhesion reflect the presence of interlinks and rearrangements that may result in fluctuations in the value of μ.

Fig. 3 reveals that the values of μ increased with increases in the annealing temperature. Because plastic deformation occurred along the scratch path and in the final form, we suspect that the increased value of μ was due to the failure of adhesion being the predominant scratch mechanism. Scratch techniques are important tools for measuring the effects of adhesion and delamination (i.e., mechanical properties) on the mechanical response. In early reported work [14], misfit dislocation segments of SiGe layers were measured during annealing; the interaction of propagating misfit dislocations with crossing ones may lead to blocking or cross-slip in different glide systems. From Wu et al. [15], although the abrasion trends of line profiles are displayed at the same ramped force, the scratch surface of the Si–Ge superlattice exhibited rather more brittle behavior than that of the Si/SiGe SLS. The elastic/plastic contact translation of Si/SiGe SLS is stable to observation for a ramped force of 2000 μN [15].

Herein, the elastic/plastic contact translation of the Si–Ge superlattice occurred in different situations. We suggest that the regions of pile-up are recorded in the middle tracking line more with annealing specimens than in RT specimens (Fig. 1); the friction traces were slightly increased after 25 s and exhibited a raised curve in each case (Fig. 2). We monitored the adhesion and delamination after moving the tip of the indenter backward with the release and build-up of force [16]; thus, we observed slightly piled-up scales and small sawtooth features for the artificial scratch for the case of a high load.

Our findings suggest a scenario involving initial dislocation to form a serial nucleation seed followed by a high-density dislocation at the interface of the bulge edge. Interdiffusion of the Si–Ge superlattices occurred at the elevated temperatures. The nanotribological behavior of the Si–Ge superlattices was evidenced using ramped sliding (Fig. 1), standard oscillation events, and response friction coefficients (Fig. 2 and 3). Therefore, annealing of Si–Ge superlattices makes them more prone to plastic deformation, resulting in higher values of μ than those obtained prior to annealing. The effect of the adhesion force on the nanomechanical damage of the Si–Ge superlattices depended on restricted dislocation movement; the critical force was also dominant in the pile-up mechanism.

4. References

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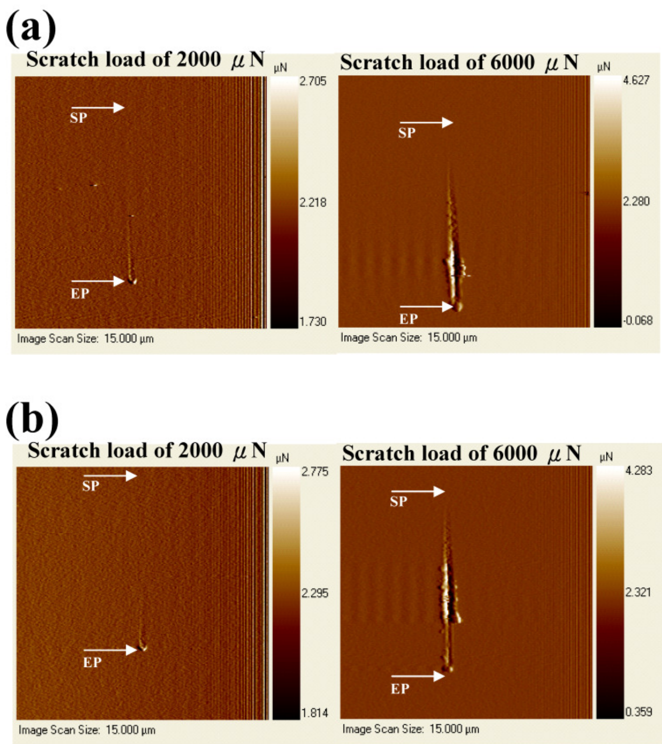
5. Conclusions

Si–Ge superlattices can be obtained using UHVCVD methods. From sliding scratch examinations, we found that the deposited Si–Ge superlattices featured marked periodicity (waviness) in their line profiles when treated under a ramped force of 6000 μN; such waviness was not obvious for the same samples subjected to a ramped force of 2000 μN. The values of μ of the Si–Ge superlattices that had been recorded at RT, annealed at 400, 500, and 600 °C were 0.09, 0.09, 0.11, and 0.11, respectively, when tested at 2000 μN and 0.095, 0.096, 0.104, and 0.102, respectively, when tested at 6000 μN. For all applied loads, the value of μ increased after annealing, because interdiffusion of the combined Si–Ge interfaces restricted dislocation movement. The Si–Ge superlattices were more brittle to pure elastic contact after thermal treatment than the specimen at RT.

6. Chart arrangement



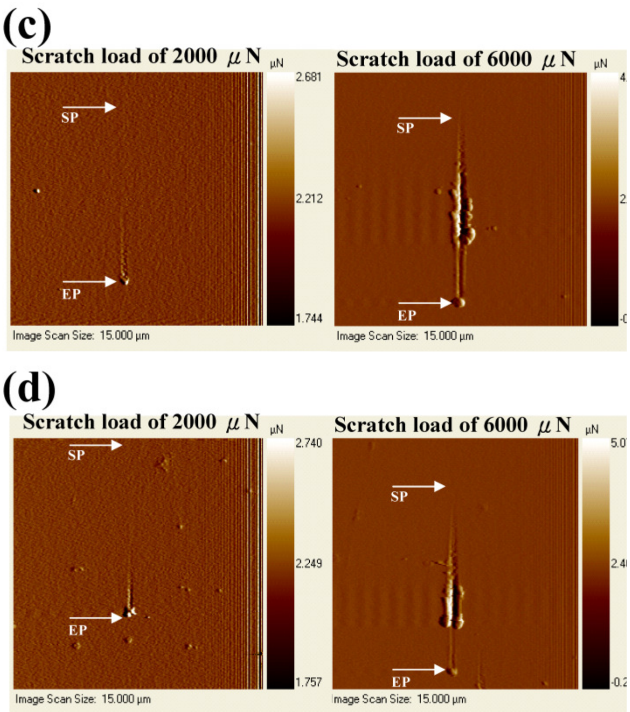
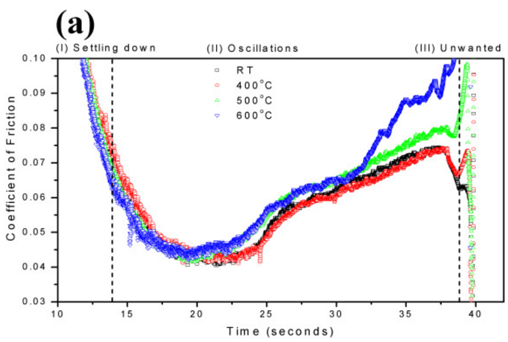


Fig. 1 3D AFM images of the surfaces of the Si–Ge superlattices and the features of the nanoscratch traces zone after annealing (a) RT, (b) 400, (c) 500, and (d) 600 °C. All of these samples had been subjected to ramping loads of 2000 and 6000 μN. We observe pure elastic contact dominated at the low ramped force (2000 μN), the annealed samples exhibited more pile-up near the middle of the sliding line (6000 μN) than did the specimen at RT.



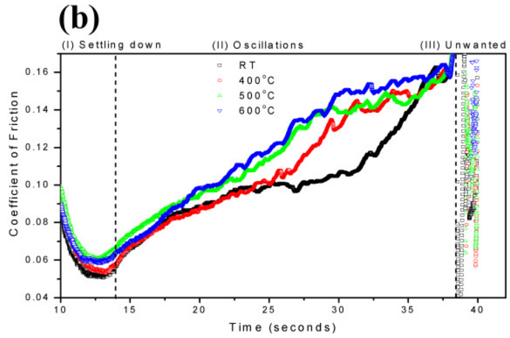


Fig. 2 Typical profiles, determined under ramping loads of (a) 2000 and (b) 6000 μN, of the coefficient of friction with respect to the scratch duration for Si–Ge superlattices that had been subjected to thermal annealing at RT, 400, 500, and 600 °C. The increasing curve is ex-situ to the artificial deformation response during the lateral force load; the possible presence of the elastic/plastic contact translation state is noted.

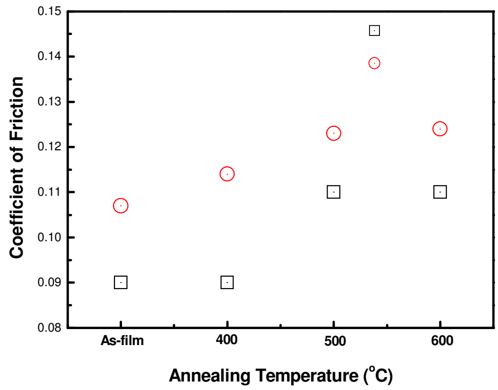


Fig. 3 Distinct critical coefficients of friction of the as-deposited and thermally treated Si–Ge thin films. Scratch techniques are important tools for measuring the effects of adhesion and delamination on the mechanical response.