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High-temperature microhardness of SiGe epitaxial layers grown on Ge and Si substrates

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Abstract
The influences of the composition of Si_xGe_{1-x} epitaxial layers, grown on Ge and Si substrates, on the microhardness and length of the dislocation rosette arms formed around indenter marks (at the homological temperatures $0.5T_{melt}$ of the corresponding alloys) have been studied. For Si_xGe_{1-x}/Ge ($0 \leqslant x < 0.15$) and Si_xGe_{1-x}/Si alloy ($0.85 < x \leqslant 1$) heterostructures, a non-monotonic dependence of the parameters investigated on the composition of the solid solution was observed. The most probable reason for these effects is the hardening at a certain composition that occurs in solid solutions.

1. Introduction

The features of the dislocation structures of epitaxial heterostructures are determined by the relationship of the plasticity of the layer to that of the substrate, and also by peculiarities of the movement and interaction of individual dislocations in epitaxial layers (EL) [1]. EL mechanical properties over a wide range of temperatures may be investigated by measurement of their high-temperature microhardness. Research on the dislocation rosettes formed around an indenter mark allows determination of the dislocation mobility in EL. In spite of its promise, investigations on the microhardness of Si_xGe_{1-x} alloy EL are rather few and they are limited to room temperature [2, 3]. 'Hot' microhardness and the structure of the dislocation rosettes formed during testing in heteroepitaxial layers of Si_xGe_{1-x} solid solutions were investigated in the present work.

2. Investigation technique

EL of Si_xGe_{1-x} solid solutions have been grown by MBE. The EL thickness was 1–2 μm. Ge(111) monocrystalline wafers were used as substrates for growth of Si_xGe_{1-x} ($0 \leqslant x < 0.15$) EL and Si(001) monocrystalline wafers—used for growth of Si_xGe_{1-x} ($0.85 < x \leqslant 1$) EL.

Microhardness measurements were carried out on an automatic microhardness tester 'Toyoseike' (Japan) with a Vickers indenter, at the homologous temperature equal to $0.5T_m$ for the corresponding alloys. As the temperature at which the contributions of the plastic deformation to the mechanical properties of the material become sufficiently different for different materials and show sufficient dependence on the melting temperature, we chose as our research temperature $0.5T_{melt}$; i.e., 330 °C for Si_xGe_{1-x}/Ge ($0 \leqslant x < 0.15$) epilayers and 570 °C for Si_xGe_{1-x}/Si ($0.85 < x \leqslant 1$) epilayers. The samples were heated at a given temperature by a special pre-arranged assembly, placed directly on the table of the microhardness tester under the indenter. The control of the sample temperature was carried out by thermocouples. Before the measurement, the samples were held at the required temperature for 15 min and then ten marks at the loading of 5 g were made. The indenter was in contact with the sample for 30 s. The choice of the load was such that the depth of the indenter penetration into the epilayer should not exceed 30% of its thickness. As follows from the results of the paper [4], in this case it is possible to neglect the influence of the substrate–layer interface and the substrates. The resulting accuracy of the microhardness measurements was 5%.

The dislocation rosettes around the indenter marks were revealed by etching Si_xGe_{1-x} ($0 \leqslant x < 0.15$) EL in an etchant based on $K_3Fe{\cdot}(CN)_6$ and KOH for 2 min and Si_xGe_{1-x} ($0.85 < x \leqslant 1$) EL in the etchant HF:0.5M CrO_3:H_2O = 4:1:1.5 for 15 s.

The diagonals of the indenter mark coincided with the directions [110] and [$\bar{1}$10]. For the length of the dislocation arms, we took half the distance between the two centres of the outer dislocation pits of the particular beam. For each mark, the average value of the lengths for two beams in two directions was calculated. Measurements were carried out on five marks and the lengths of the dislocation arms were defined as average values for each series of marks.

3. Experimental results

For all epilayers of Si_xGe_{1-x}/Ge ($0 \leqslant x < 0.15$) solid solutions investigated, diagonal cracks around the indenter marks are observed. The crack formation can be attributed to the relatively high fragility of alloys with such compositions and/or the tensile deformation of epilayers grown on Ge substrates. Probably both of these factors have some influence. In epilayers of Si_xGe_{1-x} ($0.85 < x \leqslant 1$), crack formation was not observed as a rule.

The dependences of the microhardness upon the composition for the EL investigated are presented in figure 1. It is obvious that the characters of the microhardness changes are not monotonic and are similar for the two ranges of composition. With increase of the content of the second component in the solid solution (Si for Si_xGe_{1-x}/Ge alloys ($0 \leqslant x < 0.15$) and Ge for Si_xGe_{1-x}/Si alloys ($0.85 \leqslant 1$)), the microhardness value first rises and then sharply falls, and then again grows smoothly at $x > 0.035$ and $x < 0.91$ (respectively). Maxima in the composition dependences are observed at $x = 0.025$ and 0.978 (respectively).

Typical dislocation rosettes formed around the indenter marks in epilayers of Si_xGe_{1-x}/Ge and Si_xGe_{1-x}/Si are represented in figures 2 and 3. Dislocation rosettes formed in Si_xGe_{1-x} EL consist of the central core and arms going in $\langle 110 \rangle$ directions. It is obvious that the characters of dislocation rosettes are different in layers of different composition of the solid solutions grown on the different substrates and depend upon the solid solution composition. The lengths of the dislocation arms in the solid solution layers are much less than in substrates of the corresponding pure components. In Si_xGe_{1-x}/Si epilayers, wide dislocation arms are observed, while in Si_xGe_{1-x}/Ge epilayers, they are narrow (compare figures 2 and 3).

Dependences of the dislocation arm length upon composition in the EL investigated are represented in figure 1 also. The concentration dependence of the dislocation arm length in epilayers, just like the corresponding dependence of the microhardness, has a non-monotonic

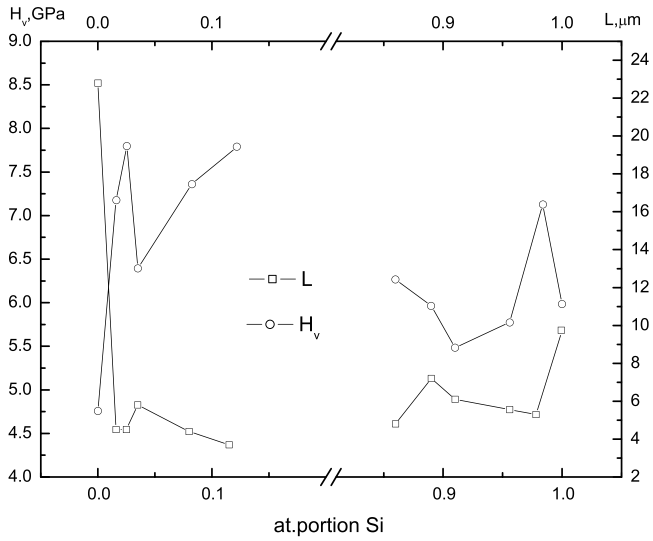
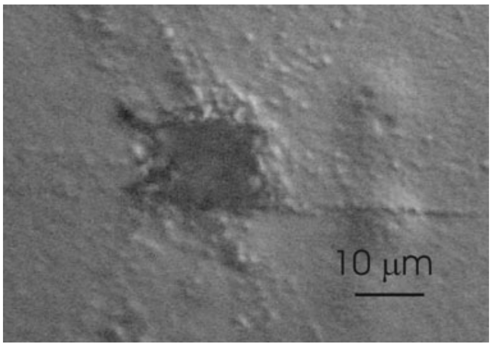


Figure 1. The dependences of the microhardness and lengths of dislocation arms upon the composition of solid solutions of Si_xGe_{1-x}/Ge and Si_xGe_{1-x}/Si for EL at the same homologous temperature $(0.5T_{melt})$.

(a)



(b)

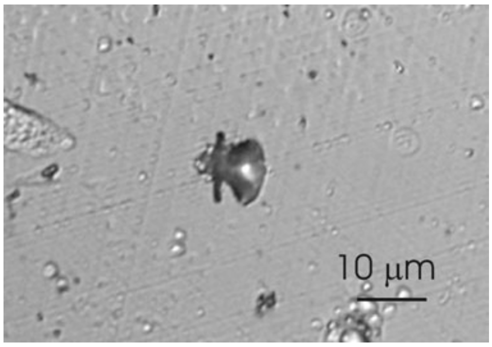


Figure 2. Typical dislocation rosettes formed around the indenter marks in epilayers of Si_xGe_{1-x}/Ge at 330 °C: (a) Ge; (b) Si_xGe_{1-x}/Ge ($x = 0.035$).

character. With increasing concentration of the second component in the solid solution, the lengths of the dislocation arms at first sharply fall, then increase a little and, subsequently, again gradually decrease for $x > 0.035$ and $x < 0.91$ (respectively). There are two well defined minima at $x = 0.025$ and 0.978 in the concentration dependence of the dislocation arm length. The positions of these minima coincides well with the positions of the maxima in the curves for the concentration dependence of the microhardness.

4. Discussion

The results obtained testify that at rather low contents of the second component in Si_xGe_{1-x} epilayers, non-monotonic changes of the microhardness and dislocation arm length formed around the indenter marks are observed. In both cases, at $x = 0.025$ for Si_xGe_{1-x}/Ge

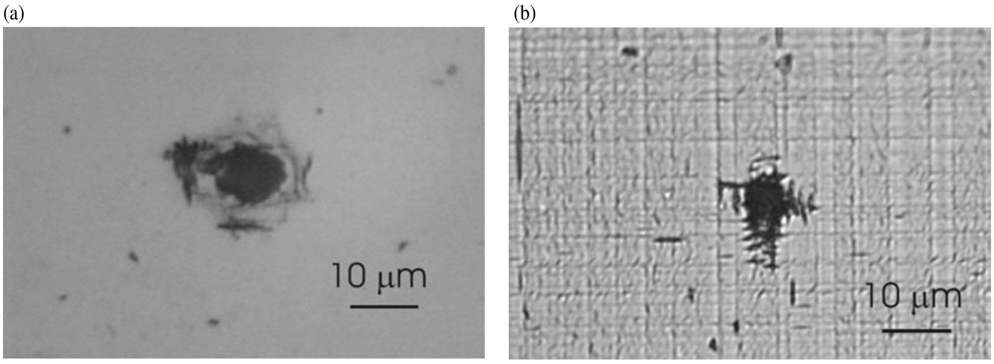


Figure 3. Typical dislocation rosettes formed around the indenter marks in epilayers of $Si_x Ge_{1-x}$/Si at 570 °C: (a) Si; (b) $Si_x Ge_{1-x}$/Si ($x = 0.11$).

heterostructure and at $x = 0.978$ for Si_xGe_{1-x}/Si heterostructure, extreme values of the measured characteristics are observed: the dislocation arm lengths are minimal at the maximal values of the microhardness. These results correlate well with the results given by us previously following an investigation of the features of dislocation structures in appropriate heterocompositions [5, 6]. In that case, the concentration dependences of the densities of inclined dislocations and misfit dislocations in EL had non-monotonic character; also, the extreme values of the measured parameters were observed at the same compositions of the solid solutions.

In papers [2, 3], microhardnesses of polycrystalline ingots and epilayers of Si_xGe_{1-x} over a wide range of composition were investigated at room temperature. In both cases, monotonic concentration dependences were observed. The authors of [4] connect the observed changes of the microhardness with changes of the force of interatomic interaction of the solid solution components. It is necessary to pay special attention to the fact that these results were obtained at room temperature, at which the contribution of the plastic deformation to the mechanical properties of solid solutions is small.

The situation is changed considerably for high-temperature measurements. With temperature increasing, the plasticity of the materials investigated grows substantially and the plastic deformation with formation of dislocations begins to play an increasing role in the microindentation processes. Direct confirmation of this is provided by the formation of the characteristic dislocation rosettes around indenter marks. The length of the dislocation arms in this case is a sufficiently objective characteristic of the mobility of dislocations formed at indentation.

The results obtained by us indicate that in the range of solid solution compositions investigated the plasticity changes non-monotonically. For a certain range of compositions ($0 \leqslant x < 0.15$ for Si_xGe_{1-x}/Ge heterostructures and $0.85 < x \leqslant 1$ for Si_xGe_{1-x}/Si heterostructures), introduction of the second component is accompanied by hardening of the EL. The most probable reason for the effect discovered is, in our opinion, the process of spinodal decomposition observed in Si_xGe_{1-x} solid solutions. At rather small contents of the second component, spinodal decomposition causes the formation of dispersed precipitates which are effective stoppers for the movement of dislocations formed during the microindentation in EL. As a result, substantial hardening of the EL occurs. This is reflected in the increasing microhardness and reduction of the dislocation arm lengths. With increasing content of the second component in the solid solutions, the size of the precipitates formed during the decomposition increases also and they cease to play the role of effective dislocation stoppers and cease to strengthen a material. This results in decreasing of the microhardness and increasing of the dislocation arm length. The characteristics of the hardness and plasticity of sufficiently concentrated alloys ($0.035 < x$ for Si_xGe_{1-x}/Ge and $x < 0.956$ for Si_xGe_{1-x}/Si) are determined chiefly by features of the interatomic interaction of the components of the solid solution and the character of their distribution in the EL.

5. Summary

The 'hot' microhardness of EL in heterostructures of Si_xGe_{1-x}/Ge ($0 \leqslant x < 0.15$) and Si_xGe_{1-x}/Si ($0.85 < x \leqslant 1$) was investigated. For the range of solid solution compositions investigated, non-monotonic and well mutually correlated concentration dependences of the microhardnesses and lengths of dislocation arms formed around indenter marks were found. The most probable reason for the non-monotonic change of the EL plasticity is the hardening of the solid solution in a certain composition range caused by spinodal decomposition of Si_xGe_{1-x} alloys with the formation of dispersed precipitates.

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