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This final report summarizes the results of our research on the Brittle-To-Ductile Transition (BDTT) using a new approach based on a cooperative dislocation generation instability. We developed a two dimensional model of the instability, extending the well-known Kosterlitz-Thouless model into the combined regime of temperature and stress. Using this model we were able to calculate the minimum temperature at which the dislocation generation instability occurs, which we identify to be the BDTT. Monte Carlo simulations were also carried out on a crystal of finite size to check the model predictions and to examine how various parameters evolve close to the instability. Experiments were performed employing a homogeneous stress field in dislocation-free Si crystals (to avoid the complexities associated with Frank-Read dislocation sources). Most experiments were performed on coherent Si/Ge overlayers on Si substrates. The sample was heated while measuring the radius of curvature to determine the temperature at which interfacial dislocations are produced in the sample. It was found that, consistent with the model, there is a massive dislocation instability at about 500C, depending upon the Ge content (which determines the stress). Similar results were obtained for three point bending experiments.

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Final Report on:

A NEW MODEL FOR THE BRITTLE-TO-DUCTILE TRANSITION BASED ON A COLLECTIVE DISLOCATION GENERATION INSTABILITY: THEORY AND EXPERIMENT

by
V. Vitek, D. P. Pope and M. Khantha

TABLE OF CONTENTS

ABSTRACT

This final report summarizes the results of our research on the Brittle-To-Ductile Transition (BDTT) using a new approach based on a cooperative dislocation generation instability. We developed a two dimensional model of the instability, extending the well-known Kosterlitz-Thouless model into the combined regime of temperature and stress. Using this model we were able to calculate the minimum temperature at which the dislocation generation instability occurs, which we identify to be the BDTT. Monte Carlo simulations were also carried out on a crystal of finite size to check the model predictions and to examine how various parameters evolve close to the instability. Experiments were performed employing a homogeneous stress field in dislocation-free Si crystals (to avoid the complexities associated with Frank-Read dislocation sources). Most experiments were performed on coherent Si/Ge overlayers on Si substrates. The sample was heated while measuring the radius of curvature to determine the temperature at which interfacial dislocations are produced in the sample. It was found that, consistent with the model, there is a massive dislocation instability at about 500°C, depending upon the Ge content (which determines the stress). Similar results were obtained for three point bending experiments.

INTRODUCTION

A new mechanism of dislocation generation that can become active suddenly above a 'critical' temperature was proposed in our research carried out under this grant. This mechanism is a thermally-driven, stress-assisted *cooperative instability* of *many* dislocation loops (dipoles in two dimensions) that leads to an outburst of dislocation activity [1-5]. The most significant outcome is that it can give rise to a transition from brittle to ductile behavior. The brittle-to-ductile-transition (BDT) is a ubiquitous phenomenon in crystalline materials, the understanding of which is of principal importance for all materials, owing to the catastrophic consequences of brittle failures in engineering components at temperatures below the brittle-to-ductile-transition-temperature (BDTT). An important characteristic of the cooperative mechanism is that it leads naturally to a sudden dramatic increase of the fracture toughness in a narrow range of temperatures (5-50°K) spanning the transition from brittle to ductile behavior in agreement with observations (see, for example, [6-12]). Moreover, this mechanism can operate in any material. Therefore, while physical properties of a given material will determine the specific temperature at which the BDT occurs, as well as the magnitude of the associated increase of the fracture toughness, the fundamental physical process underlying this transition is independent of the material. Examples of other phenomena which exhibit a dramatic surge in dislocation activity above a critical temperature include the yielding of dislocation-free whiskers [13-17] and the generation of misfit dislocations in epitaxial thin films at critical combinations of thickness, misfit strain, and temperature [18-20]. The process of dislocation generation is not well understood in both these phenomena. We have proposed and demonstrated in this program that the cooperative dislocation generation mechanism is relevant to both phenomena.

In the framework of the research supported by AFOSR during 1995-1997, the two-dimensional (2D) version of the model of the cooperative plastic instability was developed. An analytical description of a collection of interacting dislocation dipoles at finite temperatures in a loaded solid was developed using dislocation theory and statistical mechanics. In spite of the limitations arising from the 2D description, the model predicts quite well the critical temperatures above which the metallic and semiconductor whiskers deform suddenly and a variety of materials undergo the BDT. These advancements are described in more detail below. A preliminary Monte Carlo simulation that demonstrated this instability on a finite 2D square lattice was also performed. Accompanying experimental studies were aimed to provide a direct experimental evidence for the proposed mechanism. This was done by studying the temperature dependence of misfit dislocation generation in epitaxial Si/Ge single crystalline thin films on single crystalline Si substrates [21] and by the onset of plastic deformation in dislocation-free single crystalline Si beams. While an unambiguous proof of the proposed mechanism has not been yet provided, both experiments provide data that strongly support it: a massive increase in dislocation density over a very short time occurring at a critical temperature in a nearly dislocation-free material. (More details are given in the experimental section of this report.)

COOPERATIVE PLASTIC INSTABILITY MECHANISM

It is well known that the thermally activated nucleation of dislocations is extremely difficult to achieve in most materials at finite temperatures under applied loads which are well below the theoretical shear strength. Thermal fluctuations can only nucleate dislocation loops of atomic sizes which have finite formation energies in the range 0.1-2.0 eV in most materials. A formidable activation energy (usually > 10 eV) is needed to expand a small dislocation loop to the critical radius beyond which the dislocation can glide freely. Thus, even at high temperatures close to melting, thermally activated generation of glissile dislocations is considered improbable.

Kosterlitz and Thouless [22, 23] were the first to propose that unstable collective dissociation of many dislocation dipoles can occur in a two-dimensional (2D) solid above a critical temperature even in the absence of applied loads. In the original formulation, this critical event was identified with melting and/or formation of the hexatic phase in 2D systems [22-26]. The Kosterlitz-Thouless (K-T) model has since been employed in analyses of a variety of transitions which can be considered as mediated by dislocation-like defects (see e. g. [27-29]). The model advanced in our studies is based on the same physical principles. But, the important distinction is that the critical event corresponds to true plastic deformation if the effect of external loading is incorporated into the model. Such deformation then occurs suddenly at and above a critical temperature which is, however, much lower than the melting temperature. The physical reasons for this phenomenon are summarized below.

Atomic-size dislocation loops can form by spontaneous fluctuations at finite temperatures due to their small formation energies. The density of loops of radius r at a temperature T is proportional to the Boltzmann factor, $\exp[-H(r)/k_BT]$, where H(r) is the formation enthalpy of the loop and k_B, the Boltzmann constant. Under applied loads, the formation enthalpy is reduced by an amount equal to the work done by external stresses. Consequently, the density of atomic-size loops at a given temperature is higher in a loaded solid. The presence of one or more dislocation loops in a solid at finite temperatures influences the formation of subsequent dislocation loops in the solid due to the interactions between the dislocations. This is the main principle underlying the K-T model and our mechanism. The manner by which the dislocation loops influence the nucleation of further loops is similar both in the presence and absence of applied loads. However, the larger density of dislocation loops helps to enhance this influence in a loaded solid. The interactions between the dislocations in a loaded crystal at finite temperatures can be described using linear elastic theory and statistical mechanics. This is the procedure adopted in the Monte Carlo simulation. It is however possible to describe the same process using a simple mean-field approach that has been shown to be completely equivalent to the statistical mechanics based theory. We adapt the mean-field approach in the theory described below.

The self-energy of a dislocation loop in a medium depends on the stress-strain relations in this medium. In the absence of dislocations, or when the dislocations are present but cannot move, the stress-strain relations are linear and determined by the elastic moduli of the medium; the self-energy of a single dislocation loop is proportional to these elastic moduli. When dislocation loops are present in the medium and can glide, the corresponding stress-strain relations remain linear if the glide is not extensive, but are determined by *effective moduli*. These moduli now relate the stresses and *total strains*, made up of elastic and plastic components. Due to the net expansion of

dislocation loops under applied loads, the effective moduli decrease with increasing density of the loops.

This treatment represents a mean field approximation to the response of the medium with dislocations to external loading. The stress-strain relationship is formally the same as in the purely elastic case and, therefore, the dislocation stress field has the same functional form but it is now proportional to the effective moduli. Similarly, the self-energy of a dislocation loop formed in a medium containing other dislocation loops is also proportional to these effective moduli and consequently, it is lower than in the elastic case. In other words, the energy of a dislocation loop is determined not only by the elastic properties of the medium defined in the absence of dislocations but is indirectly affected by the presence of all other dislocation loops. It must be emphasized once more that this energy reduction arises due to the effective, mean change of the stress-strain relation in the medium invoked by the glide of the dislocation loops.

At low temperatures, the difference between the effective and elastic moduli is very small and the self-energy of dislocation loops differs only marginally from the sum of the self-energies of individual loops. At a constant applied load, as the temperature increases, the probability of formation of dislocation loops increases, which in turn, increases the density of dislocation loops. Concomitantly, the plastic strain in the medium increases with temperature. The ensuing decrement of the effective modulus and, hence, the self energy, provide a cooperative feedback for the further increase of the density of loops. The entropy of many loops is also much larger than that of a single loop (which is practically negligible [30]) owing to a significant enhancement of the configurational entropy. Thus, as the temperature increases, the total enthalpy of the loops is reduced while their entropic contribution to the free energy is augmented. The total free energy can then become negative at a temperature well below the melting temperature when the applied loads are large. At this point a cooperative unstable expansion of many loops ensues and gives rise to a large increase of the density of mobile dislocations. This corresponds to the macroscopic yielding of the material. In the absence of applied loads, this instability occurs just below the melting temperature and corresponds to the Kosterlitz-Thouless transition [22, 25]. The only material parameters that enter into the model are those which determine the energy of a dislocation dipole or loop, namely, the Burgers vector (b), shear modulus (μ_0), Poisson ratio (ν), dislocation core energy, inner elastic cut-off radius (r_0) and the shear stress (σ) acting on the dislocations [4, 5].

RESULTS OF THEORETICAL STUDIES

Two dimensional model of the cooperative plastic instability

We developed the model of the cooperative plastic instability in two dimensions (2D) based on the collective dissociation of many dislocation dipoles under large homogeneous stresses. While the principles of the mechanism are similar for dislocation dipoles and dislocation loops in three dimensions (3D), the analysis of the 2D model was more tractable and followed directly from the K-T model [22, 25]. Results of these studies have been published [4, 5] and here we summarize the important findings.

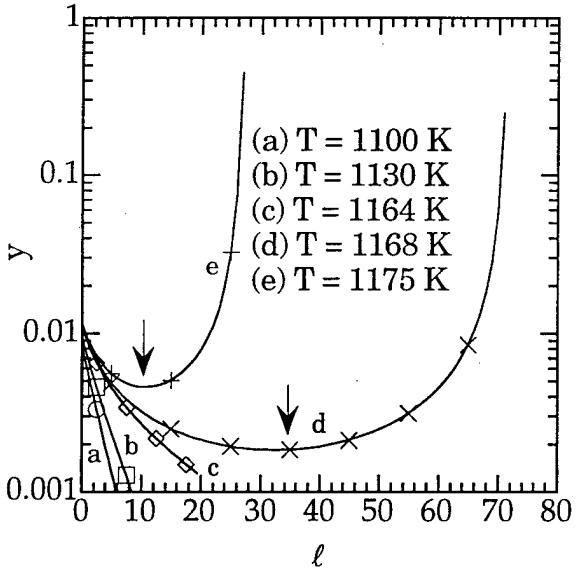

Fig. 1. The probability of formation of dipoles, y, as a function of ℓ, related to the dipole width.

The main result of the model is seen from Figure 1 which shows the dependence of the probability of formation of the dipoles (y) on their width for various temperatures. Here, y, is plotted as a function of $\ell = \ln(r/r_0)$, where r is the width of the dipole and r_0 the inner cut-off radius. The numerical values were obtained using the material parameters of silicon for a homogeneous applied shear stress of 3 GPa [4]. At low temperatures, only dipoles of atomic sizes are formed with a significant probability, larger than 0.1%, and this probability rapidly decreases with the size of the dipole. This behavior is seen for all temperatures less than and including T = 1164 K, curves a-c. However, above 1164 K the behavior is dramatically different. While the probability of formation of dipoles of larger widths initially decreases, it reaches a minimum value at a critical size (indicated by vertical arrows in Fig. 1) and thereafter this probability increases dramatically.

At T = 1175 K, the critical size for which the probability is minimum is considerably smaller than at T = 1164K and many dislocation dipoles which are initially of atomic sizes can expand and dissociate into free dislocations beyond this critical size.

This distinct change in the behavior of the probability of dipole formation in a narrow range of temperatures, close to 1164K, is a direct consequence of the cooperative reduction of the self energy and the associated increase of the entropy of many dislocation dipoles. The density of mobile dislocations increases dramatically above 1164K while it is negligible below this temperature and thus this temperature can be regarded as the critical temperature, T_c. For the same material parameters used in Fig. 1,

the instability occurs at T_c = 1943 K when no stress is applied and thus the critical temperature is substantially lowered by the applied stress. However, a large stress, of the order of a few percent of the shear modulus, is needed to reduce the critical temperature to approximately half of its zero-stress value. Hence, the dislocation nucleation mechanism proposed is expected to operate only in situations where Frank-Read sources are not present or when the stress level reaches a high value before significant plastic deformation occurs.

We have applied the model to two cases where large stresses occur, namely, the deformation of nearly dislocation-free material and the generation of dislocations in the vicinity of crack tips. In the former case our model is appropriate for whiskers or semiconductor-grade silicon in which the density of pre-existing dislocations is negligible. In the latter case, the constant stress assumed in the model has been identified with the local stress near the crack tip when the Griffith criterion for brittle crack propagation is satisfied. Below T_c, the cooperative instability does not occur and, instead, crack propagation takes place because there is insufficient dislocation activity to blunt the crack. At T_c massive dislocation generation occurs which completely precludes crack propagation. Thus, T_c for this stress level is identified with the BDTT. The predictions of the model are compared with observations in Table 1 for the yielding of whiskers and in Table 2 for the BDTT [4, 5].

There is a good qualitative and quantitative agreement between the predictions and observations in both cases. Moreover, the temperature above which whiskers deform is in the same range as the BDTT (especially for crystals other than fcc) which suggests that the process of dislocation generation is similar in the two cases.

Table 1. The critical temperature for yielding in whiskers. μ_0 is the shear modulus and σ is in the range of the measured applied stress at the onset of plastic deformation.

Material	μ_0 (GPa)	σ (GPa)	T_c (K)	Observations on whiskers
Cu	40.8	0.8	53	Deforms at 300K [14]
Ag	25.6	0.5	33	Deforms at 300K [14]
Fe	69.3	4.0	666	Fractures at 300K [13]
Zn	38.3	0.2	165	Deforms above 160K [15]
Si	60.5	4.0	908	Deforms above 900K [17]

Table 2. The BDTT for different materials compared with the calculated critical temperature T_c. μ_0 is the shear modulus and σ the stress ahead of the crack.

Material	μ_0 (GPa)	σ (GPa)	T_c (K)	BDTT (K)
Si	60.5	4.0	908	800-1200 [6]
TiAl	70.0	4.0	1090	973 [7]
NiAl	52.0	4.0	1058	700 [12]

Monte Carlo simulation of the cooperative plastic instability

In parallel with the theoretical developments we carried out Monte Carlo (MC) simulations of the cooperative instability in 2D. This calculation was made for a square lattice with elementary dislocation dipoles of widths equal to the nearest neighbor spacing as the primary entities [31]. The dipoles interact via the stress fields associated with them and a uniform shear stress is applied to the system. The Metropolis algorithm was used in the MC calculations with elementary steps corresponding to the creation and annihilation of nearest neighbor dipoles followed by expansion and contraction processes to enable the formation of dipoles of other separations. The simulations were carried out at various temperatures.

In the absence of interactions between the dislocation dipoles, nearest-neighbor (nn) dipoles have the highest probability to form and this results in a density given by the Boltzmann distribution, $\exp[-H/k_BT]$, where H is the formation enthalpy of the nn dipole. However, when the interaction between the dipoles is taken into account, both the density of nearest-neighbor dipoles and the combined density of all dipoles (nn + second nn + third nn + ...) are larger than the corresponding Boltzmann probabilities. This is shown in Figure 2 where the calculated density of dipoles per lattice site is plotted as a function of the inverse temperature, 1/T in dimensionless units. At low temperatures the densities of nn dipoles and of all dipoles are both substantially larger than the values predicted by the Boltzmann distribution. This demonstrates the energy reduction achieved by interactions of dipoles which enables the formation of more dipoles at any temperature. The theoretically determined critical temperature, T_c, is indicated by a vertical arrow. However, due to the finite size of the studied block, the dipole density does not exhibit an abrupt change at the transition temperature but varies smoothly across the transition. As seen from curve c in Figure 2, the density of all dipoles is indeed larger than the Boltzmann value for nn dipoles for smaller values of 1/T. At the same time for T appreciably higher than T_c the density of nn dipoles is slightly smaller than the density predicted by the Boltzmann distribution. This is not surprising since in this range of temperatures, the nn dipole is actually a rare configuration while dipoles of other separations are more probable.

There are many advantages of this type of MC simulation. First, it permits a direct visualization of the cooperative process, albeit on a small scale. Second, many quantities of interest, such as the number density of dipoles as a function of temperature can be extracted from the simulation data. It is usually not possible to calculate this from

theory because many configuration integrals are involved in the statistical-mechanics based analysis. Finally, it is possible to study how the instability evolves under complex stress fields by simulation while this may be difficult to carry out analytically.

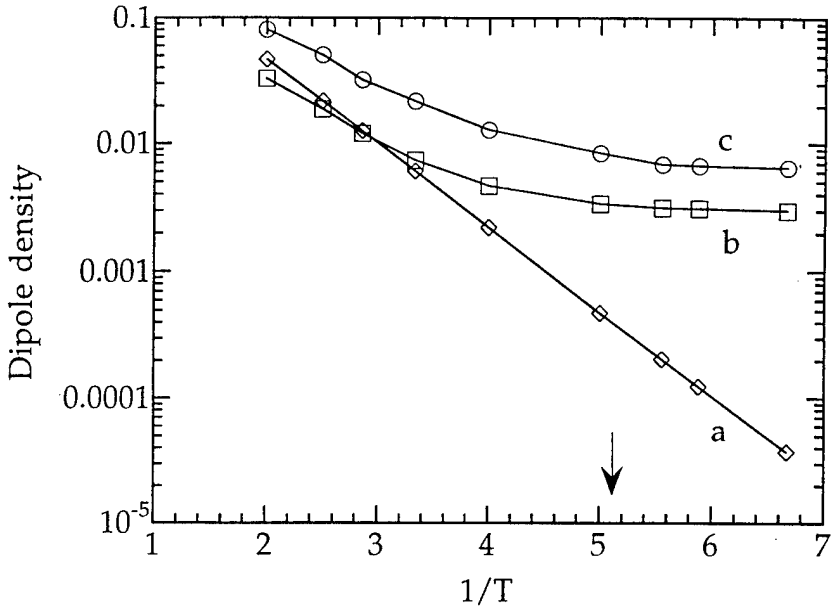

Fig. 2. The density of dipoles per lattice site as a function of inverse temperature obtained by MC simulation. Curve (a) shows the Boltzmann distribution of nn dipoles. Curves (b) and (c) show the actual nn dipole density and the density of dipoles of all separations, respectively. The simulations were carried out for a 10 x 10 lattice with periodic boundary conditions. The applied stress is equal to 2% of the shear modulus and the dislocation core energy is taken to be 1.55 eV. The arrow shows the transition temperature for an infinite crystal as predicted by the theoretical model.

RESULTS OF EXPERIMENTAL STUDIES

The model of cooperative dislocation generation has been developed at this stage for static homogeneously applied stresses and applications to cracks were made using average stresses of the crack [5]. The critical condition for the onset of the instability depends only on the temperature and the local state of stress; the particular boundary conditions do not influence the onset and, similarly, the presence of a crack is also not necessary for reaching the critical condition. Hence, the most appropriate experiments to test the model are those which concentrate on attaining the critical conditions in crack-free samples in which the state of stress (and temperature) are well-known. In fact, a pre-cracked sample would unduly complicate the interpretation. Two kinds of tests have been performed: tests on heavily stressed epitaxial Si/Ge single crystalline thin films on single crystalline Si substrates, and three point bending tests on nearly dislocation-free single crystalline Si beams. The purpose of both experiments is to apply a well characterized, intense stress field (about 1% of the modulus) to the sample at a series of known temperatures, then look for the condition of instability. In the thin film tests, interfacial misfit dislocations are produced at the instability after a short period of intense plasticity, and in the bending tests, continuous massive plastic deformation is produced.

Epitaxial films

When an epitaxial layer of a Si/Ge alloy is deposited on a single crystalline Si substrate, the lattice mismatch between the film and the substrate is initially accommodated by elastic strains in the film. These elastic strains can be as high as several percent, which translates into stresses as high as several percent of the modulus. When the epitaxial film reaches a certain critical thickness, the strains can be relieved by the generation of interfacial misfit dislocations at the substrate/alloy interface. Numerous models have been proposed over the last fifty years to predict the critical thickness as a function of the misfit strain and predict the mechanism of misfit dislocation nucleation [18, 19, 32-36]. While threading dislocations in the substrate can bend at the interface and leave behind segments of misfit dislocations, these are generally not viable sources of misfit dislocations in semiconductor epitaxy due to the very low densities of dislocations in the substrate. The interfacial plane is often not a slip plane in semiconductor films. In this case, interfacial dislocations must be produced on a different plane and then glide into the interface. The mechanism by which this takes place remains largely unclear [36].

Recently, it has been recognized that temperature plays an important role in the onset of strain relaxation in epitaxial films [34, 37-39]. For example, films can be grown at low temperatures (≤500C for $Si_{1-x}Ge_x$/Si films) to a much larger thickness than the predicted critical thickness with negligible strain relaxation, but the strain is partially or totally relieved when the films are annealed at higher temperatures (≥ 500C for $Si_{1-x}Ge_x$/Si films). It has been recognized in the past few years that surfaces of epitaxial films can roughen above a certain temperature relieving misfit strain and the resulting surface undulations provide favorable sites for the nucleation of dislocations [40-43]. These observations have renewed the debate on the mechanism of misfit dislocation nucleation and the role of temperature in the onset of strain relaxation.

Our experiments were specially designed to investigate if strain relaxation occurred suddenly above a certain temperature in films of $Si_{1-x}Ge_x/Si$ of varying thicknesses and compositions. An in-situ 'wafer curvature' experimental set-up was used which enabled us to estimate both the density of misfit dislocations and the amount of strain relief as a function of temperature. We chose a (100) oriented film on a substrate in which the dislocation density was of the order of 10-100/cm^2. In order to avoid heterogeneous nucleation at the edges of the film [36, 44], the film was produced in the form of "mesas" on the substrate with carefully angled side walls [45]. While the reason for the efficacy of this technique is not entirely understood, it is a well-known and effective means of preventing edge nucleation. The analyses of the post-growth films revealed no strain relaxation and a flat surface with no roughening.

The stresses induced in the film and substrate cause the entire assembly to bend into a spherical shape, the radius of which is a direct measure of the stress in the film. The samples were heated in-situ at the rate of approximately 5-10K per second and the radius of curvature was continuously monitored as a function of temperature using a laser scanning device. This is a one-of-a-kind apparatus that we used courtesy of Dr. Cynthia Volkert at Lucent Technologies. For samples of all compositions and thicknesses, we observed a sudden partial strain relief above a critical temperature indicative of misfit dislocation nucleation [21]. The sudden production of dislocations in the film relaxed the stress in the sample and the radius of curvature of the film increased in response to the reduction of the stress. The time rate of change of the radius of curvature is directly proportional to the mobile dislocation density through the well-known equation relating the plastic strain rate to the product of the mobile dislocation density, the Burgers vector and the dislocation velocity. The interfacial dislocation density at a given time/temperature can be estimated from the difference between the initial and the current value of the curvature. A resulting plot of sample temperature and residual strain as a function of time is shown in Fig. 3. At a certain time (temperature) the elastic strain in the film drops, at approximately 450°C in this case, indicating the onset of the plastic flow in the film. These experiments were repeated for a number of different film thicknesses and compositions (see Table 3). The data indicate that the critical temperature was near 500°C for all the films, with a scatter band of about 50°C on either side. The calculated mobile dislocation density shoots up from zero to about 10^{10} dislocations per cm^2, then drops down as the strain is relieved. The observed strain relaxation in the film was then correlated with the interfacial dislocation density using atomic force microscopy and they were found to be the same within an error of 4%.

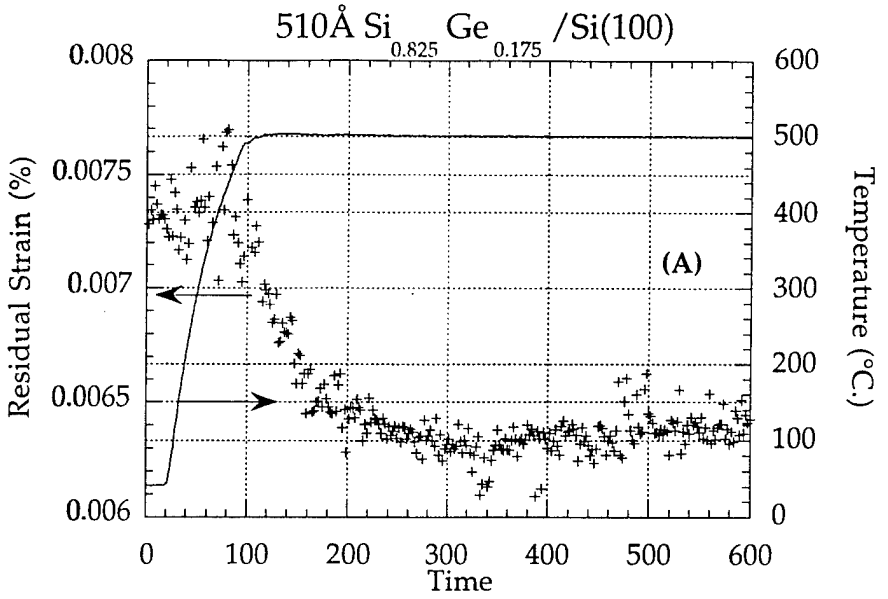

Fig. 3. The initial strain relaxation event occurs rapidly at a critical temperature, 450C in this case, indicating the formation of interfacial dislocations.

Table 3. Temperature-dependent onset of strain relaxation in epitaxial films.

Composition	Film thickness	% Strain relieved	Temperature of onset (°C)
$Si_{0.796}Ge_{0.206}$	700 Å	20.2 ± 4.6	538 ± 46
$Si_{0.796}Ge_{0.206}$	328 Å	22.1 ± 6.2	540 ± 21
$Si_{0.825}Ge_{0.175}$	510 Å	19.8 ± 4.6	500 ± 22
$Si_{0.87}Ge_{0.13}$	1850 Å	13.4 ± 9.8	504 ± 49

We consider this massive increase in dislocation density over a very short period of time in a nearly dislocation-free film to be indirect but lucid evidence for the thermally driven cooperative nucleation of misfit dislocations. Since the slip planes are not the same as the interfacial plane, a high density of dislocation loops must be generated in the film, which then grow and coalesce into a much lower density of interfacial dislocations, with spacings in the micron range, depending on the composition. The density of previously-existing dislocations is too low by many orders of magnitude to produce such a high mobile dislocation density.

There is one important question about our data: Why does the strain only relax to 80% of its original value at the critical temperature? There is more than enough stress remaining in the film to continue driving the instability, yet it stops. The relaxation only resumes again at much higher temperatures, around 700°C [39]. We do not yet have an answer to this very important question.

Bending experiments

The second set of experiments involve the application of a bending load to a small beam of Si at various temperatures. At low temperatures, below the BDTT (~600C), the beam simply cracks. At a critical temperature massive plastic flow begins, and the beam deforms into a v-shape under the point of load application. The stresses to deform the sample are approximately 1% of the modulus. Our data for the onset of plasticity in a thin sample are compared in Fig. 4 with the data obtained on Si whiskers approximately 40 years ago [17]. These data were obtained for a constant strain rate while ours were obtained for a constant stress application rate.

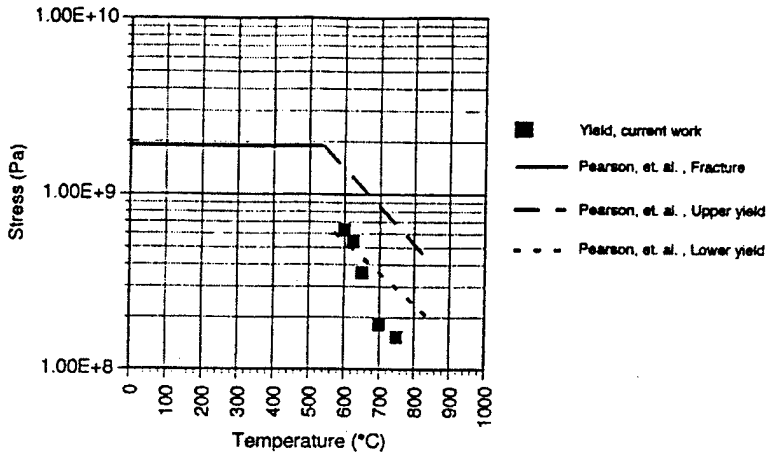

Fig. 4. Comparison of the yield point of Si beams (current work) with that of Si whiskers [17].

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