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The plasticity of indium antimonide: Insights from variable temperature, strain rate jump micro-compression testing

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ABSTRACT

At ambient temperature and pressure, most of the semiconductor materials are brittle. Traditionally, use of confining pressure via indentation or a hydrostatic confining medium has been required to study the plasticity of such brittle materials below their brittle-to-ductile transition. However, previous work has demonstrated that sample miniaturization can prevent the onset of cracking and allow plastic deformation. Here, micro-compression testing has been performed at temperatures from −4 to 300 °C *in situ* in the SEM to measure and observe the deformation of InSb. Strain rate jump micro-compression was also used at elevated temperatures to investigate the changes in strain rate sensitivity above and below the ductile-to-brittle transition. Results indicate that the flow stress follows similar trends to those observed in bulk testing with different confining media. Observations in the deformation morphology, activation energies, and activation volumes confirms that a transition from partial to perfect dislocations occurs at the ductile-to-brittle transition temperature, i.e. around 150 °C for InSb. At very low temperature (below room temperature), first results suggest another transition in dislocation nature, as observed in bulk samples.

1. Introduction

At ambient temperature and pressure, most of the semiconductor materials are brittle; at elevated temperatures, they are observed to undergo a brittle-to-ductile transition, generally situated around $0.6T_m$, where T_m is the absolute melting temperature. This is the case of the III–V compound semiconductor indium antimonide (InSb). Since the 60s, there has been extensive experimental and theoretical studies to uncover the origin of this transition [1]. All studies converge to an explanation of the brittle-to-ductile transition in terms of change of dislocation nature between the "high stress-low temperature" brittle regime and the "low stress-high temperature" ductile regime, the so-called shuffle–glide transition: the dislocation glide between two widely-spaced {111} planes ("shuffle set") in the brittle regime while they glide between two closely-spaced {111} planes ("glide set") in the ductile regime. Significantly, most atomistic simulations predict the "shuffle" regime, but there is still some controversy from the experimental point of view [1].

Traditionally, use of confining pressure via indentation or a hydrostatic confining medium [2–6] has been required to study the plasticity of such brittle materials. However, previous work has demonstrated that sample miniaturization can prevent the onset of cracking and allow plastic deformation [7,8]. This has been previously exploited using micro-pillar compression at ambient temperature to examine the deformation mechanisms of InSb at small scales [9].

Bulk techniques using elevated temperature compression in the Paterson press and indentation testing has elucidated the nature of the change in deformation in InSb [3–6]. In the ductile regime, T > 150 °C, the crystal deforms via the nucleation and motion of perfect dislocations. In theory, these perfect dislocations are dissociated (the theoretical dissociation distance is less than 10 nm, the stacking fault energy being 34–42 $mJ \cdot m^{-2}$ [10]: detailed analysis by transmission electron microscopy (TEM), has shown

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that the perfect dislocations are observed to be non-dissociated within the resolution of weak beam technique but are slightly dissociated at interaction nodes [6]. This confirms that the perfect dislocations produced at high temperature belong to the glide set [11,12]. In the brittle regime, 20 °C < T < 150 °C, wide Stacking Faults (SFs) and twin bands are observed suggesting that the crystal deformation takes place via the nucleation and glide of widely dissociated perfect dislocations or only leading partials [6], belonging to the glide set. At 20 °C, coexistence of partial and perfect dislocations has been reported: the perfect dislocations are however non-dissociated at all and exhibit cross-slip events. Below 20 °C (down to −176 °C), only perfect dislocations are observed again with footprints of cross-slip. It was then suggested that these perfect dislocations belong to the shuffle set, although the dislocation core could not be resolved [6]. All these observations conducted to the conclusion that, in bulk InSb, two transitions can be observed: at the brittle-to-ductile transition temperature (around 150 °C), there is a partial-to-perfect transition in the dislocation nature, the dislocations belonging to the glide set. At room temperature, another transition was suspected: a shuffle-to-glide transition in the nature of glide planes, necessarily associated to a perfect-to-partial transition in the dislocation nature [6].

When testing at small scales, the significance of length scale effects on mechanical properties becomes critical to comparisons between small scale test results and conventional 'bulk' results at the macro scale. For metallic samples, the effect of size on measured strength has been heavily characterized [13,14]. The dependence of flow stress as a function on sample size or diameter, d, is usually described as using a $1/d^x$ trend, where x is the so called size effect exponent. Low size effect exponents have been observed in covalent [15–17] and ionic [18,19] crystals. In these materials with high Peierls' stresses, the magnitude of the size effect exponent has been suggested to be a function of the lattice resistance on the relevant slip system [14,16], This has been experimentally demonstrated in LiF [20] to be a function of the relative contributions of the geometric strengthening and the lattice resistance to the overall strength: in InSb, the size effect has been previously observed to be small [9], but here the influence of temperature on a consistent size regime is explored.

Recent advances in *in situ* instrumentation have also enabled micro-compression techniques to extract temperature- and time-dependent deformation parameters [21,22]. Due to its well characterized brittle-to-ductile transition with temperature for bulk deformation [2], InSb is a model system for determining whether thermally activated deformation mechanisms at the micro scale correspond to macro scale behavior. Additionally, InSb's low melting point of 527 °C allows it to be interrogated over a significant fraction of its solid temperature range, which will provide insight into the high temperature behavior of other semiconductors with a similar structure but higher melting point. Here, strain rate jump micro-compressions at elevated temperature have been performed *in situ* in the SEM to measure the activation parameters of the plasticity of InSb at the micro scale at temperatures above and below the brittle-ductile transformation. Complementary TEM studies have been performed when necessary to confirm the dislocation nature in the deformed micro-pillars.

2. Materials and methods

2.1. Sample production and characterization

Bulk S-doped ($n = 4.88 \times 10^{16}$ cm^{-3}) InSb single crystalline slabs were cut out from larger crystal and chemically polished to remove defects introduced during the cutting operation that would lead to complex initial microstructure in the micro-pillars. With this operation, the slabs contain an initial density of dislocations very similar to the bulk samples previously studied, i.e. of the order of 10^{12} m^{-2}. The initial dislocation density have been measured in bulk samples and in micro-pillars by careful inspection of TEM foils extracted from the two types of samples [5,6,9]. Several crystallographic orientations have been selected but only compression along the [213] direction is reported here as this orientation favors single slip with the activation of the $a/2$ [011] (-11-1) slip system with a Schmid factor of 0.47.

Micro-pillars with diameters of ~2 μm with aspect ratios of ~3 were fabricated using a Ga ion beam at an accelerating voltage of 30 kV in Tescan Vela and Lyra FIB instruments. Initially, high currents of ~6 nA were used to mill craters around micro-pillars with larger than targeted diameters, which allow clear viewing of the entire pillar length during compression. Next, the desired micro-pillar dimensions were achieved by polishing the coarse micro-pillars with lower currents ranging from 1 nA to 300 pA, which in turn minimizes irradiation damage. The micro-pillars were imaged using a Hitachi S4800 high-resolution scanning electron microscope (HRSEM) and SEM in the Tescan Lyra FIB after FIB machining and after compression at elevated temperatures.

The TEM samples investigated were prepared by the FIB lift-out technique [23] using the same Tescan Lyra and Vela instruments. The pillars were first coated with Pt using a gas injection system (GIS) and then lifted out and attached to copper TEM grids. Final polishing of the samples was performed using a 5 kV ion beam at 1° to the lamellae surfaces. A JEOL JEM-2200FS TEM operating in scanning mode (STEM) was used to image the pillars.

2.2. Micro-compression

The system used for micro-compression is an Alemnis *In-situ* Indenter modified for high temperature operation [21] *in situ* in a Zeiss DSM 962 SEM operated at 5 kV. Room temperature tests (monotonous and cyclic compression) were performed at a strain rate varying between 4×10^{-5} and 5×10^{-4} s^{-1}. For elevated temperature testing, thermal drift in this system is minimized at each testing temperature by both temperature shift [24] and displacement drift [7] tuning measurements during pseudo load-controlled indentation. The indenter tip temperature was calibrated using the procedure described previously [24,25]. The sample temperature was varied prior to contact to match the calibrated indenter temperature, at which point both displacement drift and temperature drift during contact were at a minimum.

Sub-ambient temperature testing was conducted using a slight modification of the elevated temperature setup. The insulating heater components were swapped with conductive copper parts which were connected directly to the system frame using copper braid. Low temperature coolant (1:1 ethylene glycol and water mix) was then circulated through water-cooling channel in the system frame to cool the entire system. The sample and indenter temperature were monitored using type K thermocouples. Thermal displacement drift during low temperature testing was observed to be identical to ambient temperature conditions. For measuring temperature dependence under constant strain rate, micro-pillars were compressed at a constant displacement rate appropriate to generate a 4×10^{-4} s^{-1} strain rate for the heights of the pillars after compliance correction. The minimum temperature achieved with the current chiller was −4 °C, but further cooling to −50 °C is expected to be feasible with a more powerful commercial chiller. This is under development, and further work will include strain rate sensitivity and size effect measurements at sub-ambient temperatures.

Strain rate sensitivity was determined by performing strain rate jumps during elevated temperature micro-compressions. Strain