The Taylor relation in compression deformed Ge single crystals

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Abstract. Ge single crystals are deformed in compression at 850K and the same strain rate to various extents of strains. In each sample, the internal stress is measured through stress reduction tests and the dislocation densities by X-ray measurements. Data about these two parameters follow fairly well the Taylor-Saada relation, provided a correction term is added. It probably corresponds to dislocations which are seen by X-rays, though they do not contribute to crystal hardening.

1. Introduction

As a dislocation glides through a Germanium crystal, it has to overcome two types of “obstacles”: localized obstacles consist of the lattice resistance to glide (Peierls Nabarro force) while the others are due to long distance elastic interactions with other dislocations. Only the former ones are overcome with the help of thermal activation. They necessitate a shear stress \(\tau^*\) called the thermal stress. The second ones necessitate a stress \(\tau_i\), called the internal stress. It has been proposed \([1]\) that the applied shear stress \(\tau\) can be decomposed according to:

\[
\tau = \tau^* + \tau_i
\]  

(1)

The point of this study is to relate values of \(\tau_i\), measured through stress reduction experiments in compression deformed Germanium single crystals at one temperature \(T\) and (shear) strain rate \(\dot{\gamma}\). Then check the Taylor-Saada relation that claims that \(\tau_i\) is proportional to the square root of the dislocation density \(\rho\).

\[
\tau_i = \alpha Gb\sqrt{\rho}
\]  

(2)

There is a large amount of investigations about the plasticity of covalent crystals over the past 40 years (see reviews by e.g. \([2, 3]\)). To the best of our knowledge, preliminary work about the
determination of $\tau^*$ and $\tau$, and the origin of the internal stresses in Ge single crystals can be found in [4-6]. Similarly, the parameter $\rho$ which is usually measured through the observation of etch pits with an optical microscope at specimen surfaces, is estimated here by appropriate X-ray techniques. High resolution double crystal diffractometer dedicated for line profile analysis is used to determine active slip systems and dislocation densities along with the fluctuation of dislocation densities as a function of deformation in Ge single crystals.

2. Experimental procedures

2.1. Material
Intrinsic Ge single crystals were cut in a parallelepipedic shape by a diamond saw in the $[-123]$ single slip orientation with (1-11) and (54-1) lateral faces. The primary slip system is $[-101]$ (111). Such an orientation ensures a much larger Schmid factor on the primary system as compared to the secondary one [011] (-1-11) (0.47 and 0.38 respectively). Care was taken to remove the dislocation rich surface layers by mechanical and chemical polishing.

2.2. Mechanical testing
Stresses $\tau$, strains $\gamma$ and strain rates $\dot{\gamma}$ are resolved with respect to the primary slip system. The compression specimens were deformed in a computer controlled Schenck RMC 100 set up, equipped with a furnace, under a flux of He. The testing temperature was 850K at shear strain-rate $\dot{\gamma}$ of $1.6 \times 10^4$ s$^{-1}$. At the end of the test, the Ge samples were cooled under load so as to freeze the stressed dislocation structures. In an attempt to estimate the internal stresses along the monotonic curves, dip tests were performed using a procedure first proposed by [7]. At given stress along the stress strain curve, fast unloading is applied followed by a short creep experiment (15 seconds). The amount of stress decrease that corresponds to a zero creep rate equals the effective stress under these conditions [5,7]. For this purpose, computer programs have been designed for machine control and data acquisition. Details about the procedure can be found in [5].

2.3. X-ray investigations
The investigated face of the specimens is (54-1). The diffraction profiles were measured by a special double crystal diffractometer with negligible instrumental broadening. A fine focus rotating copper anode, Nonius FR 591, was operated as a line focus at 40 kV and 70 mA. The symmetrical 220 reflection of a Ge monochromator was used in order to have wavelength compensation in the lower angle region. The profiles were registered either by a linear position sensitive gas flow detector, OED 50 Braun, Munich, or by two dimensional imaging plate detectors of 100 mm height and 200 mm length with 50 $\mu$m pixel size.

3. Results
A typical stress strain curve is presented in Fig. 1. It exhibits the well known multiplication yield point (UYP) at the beginning, while normal hardening sets in after the lower yield point (LYP). Given the extremely low dislocation density in covalent crystals, intense multiplication phenomena operate at the onset of deformation. An evaluation of multiplication laws can be found in [8]. An example of the stress decomposition into effective and internal parts is illustrated in Fig. 1, showing that under the present conditions, $\tau^*$ ($\approx 6.1$ MPa) is constant along the curve, within the uncertainty on the measurement. This is expected for the Peierls stress, under such conditions. $\tau$ is an increasing function of strain, due to dislocation hardening, above the lower yield point, $3.5$MPa $< \tau < 8$MPa in the case of Fig. 1.
Figure 1. Stress-strain curve, effective and internal stress along the curve. 

\[ T = 850\text{K}, \ \gamma = 1.6 \times 10^{-4} \text{s}^{-1}. \]

Two other specimens were deformed at the same temperature and strain rate up to different levels of total strain (see Table 1). The above X-ray experiments were used to measure the dislocation densities in the three specimens after deformation. Table 1 summarizes the results.

| \( \gamma_p (\%) \) | \( \tau (\text{MPa}) \) | \( \tau^* (\text{MPa}) \) | \( \tau_i (\text{MPa}) \) | \( \rho (\text{m}^{-2}) \) |
|---------------------|-----------------|-----------------|-----------------|--------------|
| 7.0                 | 10.4            | 6.0             | 4.4             | 2.6x10^{13}  |
| 20.5                | 14.0            | 6.0             | 8               | 2.0x10^{13}  |
| 30.8                | 36.6            | 6.0             | 30.6            | 6.5x10^{13}  |

4. Discussion

In an attempt to check whether the data about \( \tau_i \) and \( \rho \) of Table 1 fit the Taylor-Saada relation, they have been plotted in Fig. 2 which shows \( \tau_i \) as a function of \( \sqrt{\rho} \). Given the uncertainty on the measurement of the internal stress and especially the dislocation densities, a linear relation between \( \tau_i \) and \( \sqrt{\rho} \) is acceptable in agreement with the Taylor formula. Inspection of Fig.2 yields the following remarks:

i) The slope of the line on Fig. 2 equals \( \alpha Gb \) according to equation (2). With \( G = 60 \text{ GPa} \) and \( b = 4.0 \times 10^{-10} \text{ m} \), the slope of 7.09 Pa m measured on Fig. 2 yields a value of \( \alpha = 0.295 \). This can be compared to previous attempts at estimating the coefficient \( \alpha \). Lavrentev [9] lists measured values of \( \alpha \) in various situations. A value between 0.2 and 0.33 is reported for Cu single crystals at 300K deformed in stages I to III. Berner and Alexander [10] propose a theoretical value of \( \alpha = \frac{1}{2\pi(1-v)} = 0.23 \) for Ge single crystals and a measured one of 0.32, using the etch pit technique for density measurements. The present data are in fair agreement with these estimations, given the uncertainty of measurements.

ii) Extrapolating the straight line of Fig. 2 yields a zero value for \( \tau_i \) that corresponds to a certain dislocation density \( \rho_0 \), so that relation (2) should be written:
\[ \tau = \alpha G b (\sqrt{\rho} - \sqrt{\rho_0}) \]  

(3)

The density \( \rho_0 \) corresponds to dislocations which are seen by X-rays but do not contribute to hardening the crystal. Dislocation dipoles could be a possible type of such dislocations since they distort locally the lattice but do not create long distance stress-fields.

5. Conclusions

The two independent experimental techniques which have been used here to measure respectively the dislocation density and the internal stress in samples deformed to various extents at the same temperature and strain rate yield a relation between these parameters which strongly supports the Taylor relation, as old as 1934. The coefficient \( \alpha \) is in fair agreement with previous theoretical estimations or measurements through other means. A correction term has to be added to the formula. It corresponds very likely to dislocations that are seen by X-rays but do not contribute to crystal hardening.

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