The Mechanical Behavior of Silicon During Small-Scale Indentation

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The mechanical behavior of crystalline silicon during small-scale indentation has been studied using a Nanoindenter. Tests were performed on both p-type and n-type materials in the (100), (110), and (111) orientations at peak loads ranging from 0.5 to 120 mN. The indentation load-displacement curves exhibit two features which appear to be unique to silicon. First, at large peak loads, a sharp discontinuity in displacement is observed as the indenter is unloaded. Second, at small peak loads, a large, non-degenerate hysteresis is exhibited. Possible mechanistic origins for the discontinuity and hysteresis are discussed.

Key words: Si, mechanical behavior, indentation

INTRODUCTION

The work reported here is a part of a larger study in which the indentation load-displacement characteristics of a wide variety of materials are being characterized using the Nanoindenter. The Nanoindenter is a specialized hardness tester in which it is possible to make indents as small as a few nanometers in depth.1,2 The instrument continuously records both the indentation loads and displacements, and from this data it is possible to derive a variety of mechanical properties such as hardness and modulus.3 One of the great advantages of the technique is that it can be used to measure the mechanical properties of thin films and small volumes, and as such, the instrument can be used as a mechanical properties microprobe.4

In order to properly interpret indentation load-displacement data, it is necessary to understand the variety of mechanical behaviors and deformation mechanisms which can occur during indentation. To this end, we have, for the last three years, been characterizing and cataloging the indentation load-displacement characteristics of a large number of metals and ceramics. During the course of this work, it has become evident that the load-displacement behavior of silicon is different from that of most other materials. In this paper, we document this behavior, outline mechanisms which might explain its origins, and discuss why the behavior is observed only in silicon.

EXPERIMENTAL

The material used for the majority of the study was single-crystalline, p-type silicon with a (110) orientation. A limited number of experiments on other forms and orientations of crystalline silicon revealed no major effects of doping or orientation on the indentation load-displacement characteristics. A discussion of the behavior of amorphous silicon, which is significantly different, will be given in a forthcoming report.5

Specimens were indented using the Nanoindenter at the Oak Ridge National Laboratory. A Berkovich indentor, a triangular pyramid diamond whose depth-to-area relation is the same as that of a Vickers indenter, was used in all experiments. No attempts were made to align the indentor relative to the crystal symmetry axes.

Two basic types of tests were performed. In the first, the indenter was loaded into the specimen at a constant loading rate to predetermined peak load, \(P_{\text{max}}\), and then unloaded at the same rate to 10% of \(P_{\text{max}}\). This was then followed by a second loading/unloading cycle. Three values of \(P_{\text{max}}\) were investigated. The first, \(P_{\text{max}} = 120\) mN (12 g), is the maximum load which can be applied by the Nanoindenter. These indents were produced at a loading rate of 1500 \(\mu\)N/s. The second, \(P_{\text{max}} = 12\) mN, was achieved by programming the Nanoindenter controller to reduce the peak load by a factor of 10. At the same time, the loading/unloading rate was reduced by the same amount to assure that all the load-displacement curves consisted of a similar number of data points (the rate of data acquisition in the Nanoindenter is constant at about 2–3 data points per second). Subsequently, \(P_{\text{max}}\) and the load rate were reduced by another factor of 10 to produce a third set of indents at \(P_{\text{max}} = 1.2\) mN. In further discussion, these tests will be referred to as 2-cycle tests.

The second type of test was similar, but involved four loading/unloading cycles at faster rates. In all, six separate peak loads were investigated in these 4-cycle tests: 120, 40, 13.3, 4.5, 1.5, and 0.5 mN. The loading/unloading rate for the 120 mN indents was 7500 \(\mu\)N/s and was reduced by a factor of 3 for each successive peak load. An example of how the load
was varied with time in a typical 4-cycle test is shown in Fig. 1.

For both the 2-cycle and 4-cycle tests, a 100 sec hold period was included in the indent sequence just prior to final unloading (see Fig. 1). The purpose of the hold was to establish the rate of displacement produced by thermal expansion in the system. Even though the Nanoindenter is thermally buffered from its surroundings and the room in which it is housed is temperature-controlled to within ±0.1°C, small thermal fluctuations cause the system to expand, and the expansion is manifested as an apparent displacement in the specimen. Thermal drift becomes particularly important for small indents made over long periods of time, as was the case for some of the indents in this study. To account for it, the rate of displacement was measured during the last 80 sec of the hold period, and the displacement data were corrected by assuming that this drift rate was constant throughout the test.

RESULTS

In order to appreciate how unique the small-scale indentation load-displacement behavior of silicon really is, we first present, for the sake of comparison, data for two materials whose load-displacement curves are representative of the great majority of hard, brittle materials. The behavior of silicon is subsequently described.

Fused Silica

Typical load-displacement curves for fused silica obtained in a 2-cycle test are shown in Fig. 2. The noteworthy features are the relative smoothness of the curves, and how all the data subsequent to the first loading fall on a single curve. The significance of the latter observation is that it implies that the plastic part of the deformation occurs entirely during the first loading, and subsequent deformation is purely elastic. As the peak load and loading rate are reduced, there is virtually no change in behavior; the curves maintain their same general form, and without the aid of scales on the axes, it is difficult to distinguish a set of data obtained at a peak load of 120 mN from a set obtained at 1.2 mN.

Soda-Lime Glass

The second sort of behavior observed in hard, brittle materials is typified by soda-lime glass, and data obtained in a 4-cycle test on a simple glass microscope slide are presented in Fig. 3. In contrast to the behavior of fused silica, the loading and unloading curves following the initial loading are slightly hysteretic—the second loading curve is displaced slightly above the first unloading curve so as to form a distinct loop. However, this looping degenerates in higher cycles, so that after three of four