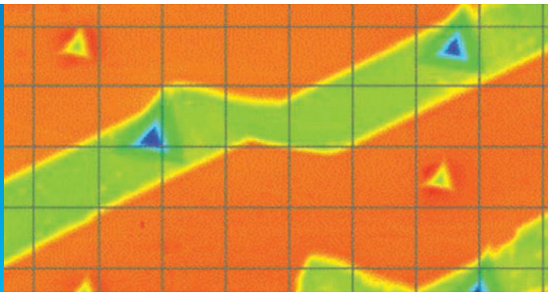
Application Note

Brittle-to-Ductile Plasticity Transition Behavior Study of Silicon using High-Temperature Nanoindentation



Introduction

Single crystal silicon (SC-Si) has been extensively used in the semiconductor industry to make micro/nano-electronic devices. The brittle-ductile transition (BDT) of single crystal silicon (SC-Si) is of great practical importance in the development and manufacture of these devices as they require many thermomechanical processing steps. Understanding the material behavior, i.e. deformation mechanisms and phase transformation, improves control and enables prediction of Si failure during thermomechanical processing. One of the thermomechanical properties of many materials is their brittleness at low temperatures, and ductility at higher temperatures. This transition occurs over a narrow range of temperature for Si, referred to as brittle-to-ductile transition temperature (TBD).

Varying the temperature during the primary mechanical testing method, e.g. fracture toughness test, is one way of measuring the transition plasticity of materials that are otherwise not easily tested on the nanoscale. Nanoindentation at elevated temperatures also provides the ability to accurately measure the nanomechanical response of Si at various temperatures up to and above TBD to evaluate the plasticity transition. This is critical for the improvement of electronic device fabrication methods. This method offers a direct measurement of fracture toughness and nanoscale material response, accurate probing and imaging of the crack length, and indentation geometry at transition temperatures.

In this application note, plasticity transition and creep behavior of a standard (100) silicon wafer has been studied using the Nano Indenter G200 laser heater system at elevated temperatures up to 500°C. The sample was loaded to the maximum load of 570mN and held for 60 seconds at room temperature (RT), 250°C and 500°C. The sample was mounted on a specially designed heating stage that uses a laser as a rapid heating method. The Nano Indenter G200 system also employs a specially designed laser-heated tip to avoid any contact thermal drift during the test and to provide stable testing conditions. Both stage and tip are equipped with separate thermocouples that read their exact temperatures. The results of the investigation in this application note show how changes in plasticity of Si at high temperatures are relevant to nanoindentation mechanical measurements. This application provides a very simple and precise procedure of studying high-temperature nanoscale transitions in different materials. It also has many potential applications in thin film high-temperature phase transformations.

Single crystal Si is a brittle material at room temperature in which cracks propagate without any appreciable plastic deformation because of the sp3 bonding and diamond cubic crystal structure of Si. Nevertheless, it exhibits the ductile behavior of metal above a specific temperature, called brittle-to-ductile transition temperature (TBD). This sudden increase in fracture toughness is attributed to a large increase in dislocation density in the microstructure and increased mobility with rising temperature. It was found that for Si, unlike metals, this transition occurs in a very narrow temperature range of 541-545°C for bulk material.[1] It was also shown that this temperature is size-dependent, for instance in SC-Si nanowires,[2] TBD reduces with sample size to 250°C.[1] Changes in the plasticity behavior are also reflected on the loading-unloading nanoindentation curves. Figure 1a-c shows the loading-unloading curves at various testing temperatures, demonstrating that increasing the temperature from room temperature to 250°C produces no pronounced difference in the maximum indentation depth or behavior of loading-unloading curves. However, at 500°C, loading-unloading indentation hysteresis displays larger deformation and penetration depth.

At room temperature, due to the Si phase transformations and their corresponding volumetric changes during loading-unloading, a distinct displacement discontinuity is observed in the curves (indicated by the arrow) in Figure 1a. This discontinuity is controlled by the amount of hydrostatic and deviatoric stress applied to components under the indenter tip, and is also highly dependent on the tip geometry. Such defective loading-unloading curves can be attributed to the formation of deep lateral cracks beneath the indenter tip. The steps observed on unloading curves are called pop-outs and are probably caused by the transition to the lower density structures associated with volumetric expansion. At higher testing temperatures of 250°C and 500°C, there is no evidence of such events and the load-penetration curves look well-behaved (Figure 1b-c).

Looking more carefully at the creep behavior of Si (dashed boxes in Figure 1a-c), it is seen that at a constant peak load of 570mN, the material underwent a large deformation over time at 500°C (Figure 1d). These curves show almost no changes in displacement into the surface during the 60 second peak holding time at room temperature and 250°C, whereas there is about a 200nm indentation at the constant peak load at 500°C. Apparently, there is a large transition in the plasticity behavior of Si from brittle to ductile during indentation at 500°C. This is lower than what was reported in the literature for bulk SC-Si which was approximately 545°C.[1] This may be due to the size effect and small-volume characterization of plasticity behavior prevailing in nanoindentation.

Material Response at High-Temperature Nanoindentation

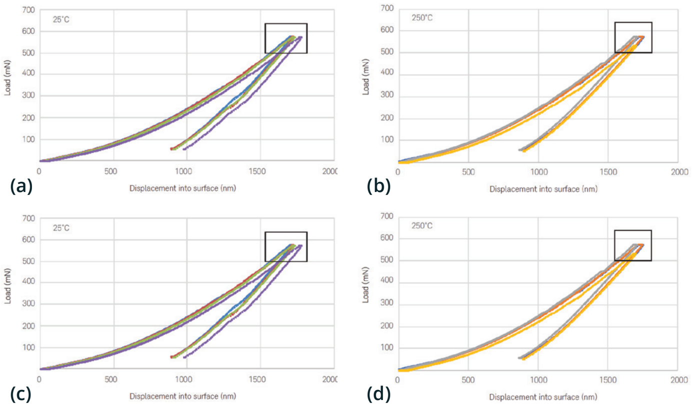


Figure 1. (a-c) Load versus displacement of Si (100) at various temperatures under elevated nanoindentation. (d) The displacement into the surface at peak load (570mN) versus holding time of the areas highlighted in the dashed boxes.

Figure 2 displays the indentation shape after unloading which was obtained by optical imaging at different testing temperatures. Clearly, at room temperature, brittle cracks, pop-outs and uniform indent are seen. At 250°C, cracks still appear during indentation, but there is no evidence of pop-outs. The indentation shape is comparable to that at room temperature. However, at 500°C, no cracks are observed. In addition, there is a distinct pile-up around the indenter tip edges with uniform indentation shape similar to what was observed in ductile metals like aluminum (Al). No pop-outs during unloading are observed either. The above evidence demonstrates the ductile behavior of Si at 500°C.

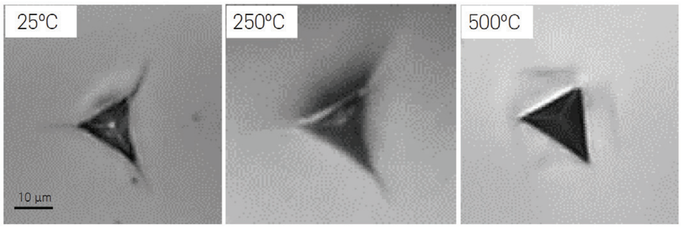


Figure 2. Optical images of indents after nanoindentation at various testing temperatures.

Conclusions

Silicon usually shatters like glass at room temperature, but shows ductile fracture like metal when the temperature is increased to 500°C. The nanomechanical response of single crystal silicon at various temperatures was studied using high-temperature nanoindentation with the Nano Indenter G200 laser heater. The brittle-to-ductile plasticity transition was expressed on the loading-unloading curves. High-temperature nanoindentation measurements provide scientists with the means to better understand the material transition behaviors and to improve thermal management at elevated temperatures.

References

1. PB. Hirsch, and SG. Roberts, The brittle-ductile transition in silicon, Philosophical Magazine A, 1991, volume 64, number 1, pages 55-80.

2. W. Kang, M. Saif, In situ study of size and temperature dependent brittle-to-ductile transition in single crystal silicon, Advanced Functional Materials, 2013 volume 23, number 6, pages 713-719.

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