

Keysight Technologies

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

Application Note

Introduction

Single crystal silicon (SC-Si) has been extensively used in 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 that they are brittle at low temperatures, but become ductile at higher temperatures. This transition occurs over a narrow range of temperature for Si, referred to as brittle-to-ductile transition temperature (T_{BD}).

Varying temperature during the primary mechanical testing methods, e.g. fracture toughness test, is one way of measuring the transition plasticity of materials that are not easily tested on the nano-scale, which is used to identify the scale-sensitivity of such transitions. Nanoindentation at elevated temperatures provides the ability to accurately measure the nanomechanical response of Si at various temperatures up to and above T_{BD} and 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 nano-scale material response as well as 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 Keysight G200 laser heater system at elevated temperatures up to 500°C. The sample was loaded to the max load of 570 mN and was 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 very fast heating method. The G200 laser heater system also uses a specially designed laser-heated tip to avoid any contact thermal drift during the test and to provide very 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 nano-scale, high-temperature transitions in different materials. It also has many potential applications in thin film high-temperature phase transformations.

Material Response at High-Temperature Nanoindentation

Single crystal Si is a brittle material at room temperature in which cracks propagate without any appreciable plastic deformation. This is because of the sp^3 bonding and diamond cubic crystal structure of Si. Nevertheless, it exhibits the ductile behavior of metal above a specific temperature, this is called brittle-to-ductile transition temperature (T_{BD}). This sudden increase in fracture toughness is attributed to a large increase in dislocation density in the microstructure and their increased mobility with temperature. It was found that for Si, unlike metals, this transition occurs in a very narrow temperature region 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], T_{BD} reduces with sample size to a couple of hundreds degree C (250°C) from what was observed in bulk material (545°C)[1]. Changes in the plasticity behavior can also be reflected on the loading-unloading nanoindentation curves. Figure 1a-c shows the loading-unloading curves at various testing temperatures. The figure shows 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 is controlled by the amount of hydrostatic and deviatoric stress components under the indenter tip, which 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, probably caused by the transition to the lower density structures associated with a 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 570 mN, 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-s peak holding time at room temperature and 250°C, whereas there is about a 200-nm 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.

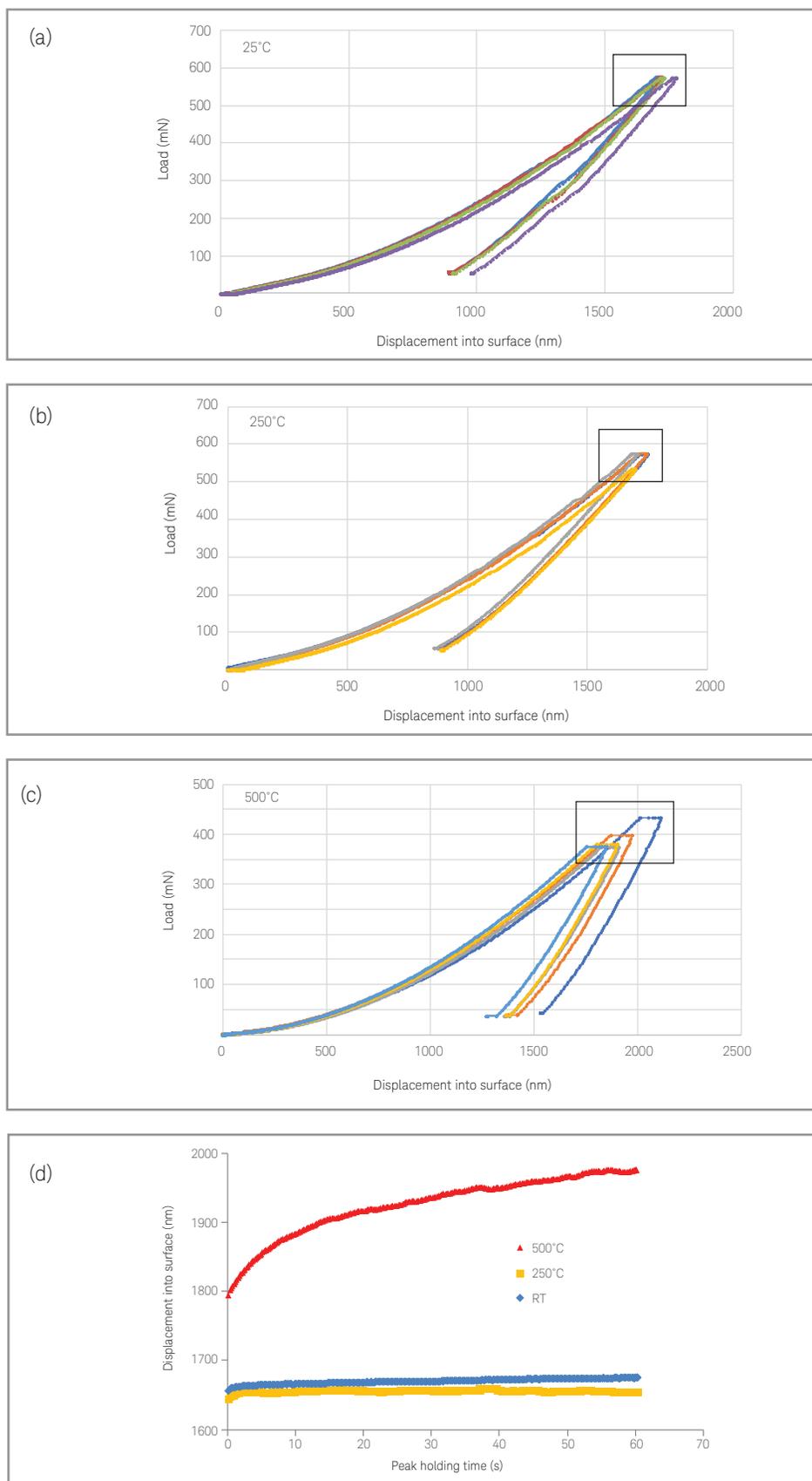


Figure 1. (a-c) Load versus displacement of Si (100) at various temperatures under elevated nanoindentation. (d) The displacement into the surface at peak load (570 mN) versus holding time of the areas highlighted in the dashed boxes.

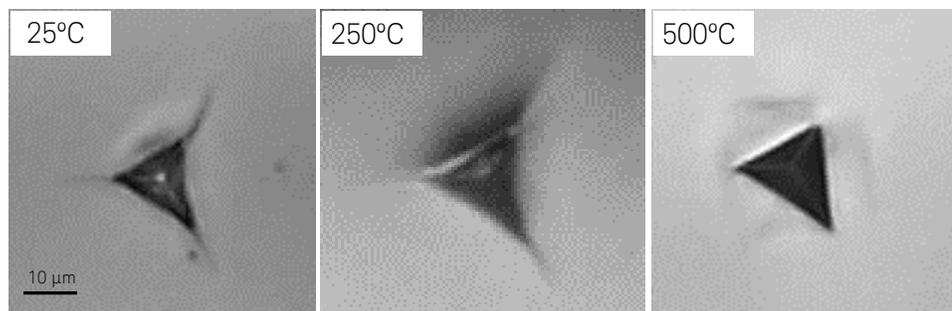


Figure 2. Optical images of indents after nanoindentation at various testing temperatures.

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 in Si at 500°C.

Table 1 compares hardness and Young's modulus measurements obtained from the aforementioned tests. Results show that although there is a decrease in the modulus and hardness of Si at 250°C, the change is not linear when increasing the temperature to 500°C. It confirms a sudden change in plasticity transition at 500°C as the modulus and hardness decrease to about 40% of those at room temperature.

Table 1. Modulus and hardness of (100) Si at various temperatures under elevated nanoindentation.

Test temperature (°C)	Mean Young's Modulus (GPa)	Std. Dev. (GPa)	Mean Hardness (GPa)	Std. Dev. (GPa)
RT	274.01	15.17	24.03	1.35
250	217.82	13.63	19.95	1.79
500	164.445	8.88	9.44	0.57

Conclusions

Silicon usually shatters catastrophically 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 Keysight 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.

Acknowledgement

The investigation of this paper was carried out by D. Bedorf and M. Knieps at the SURFACE NanoLab, Hückelhoven, Germany.

Evolving Since 1939

Our unique combination of hardware, software, services, and people can help you reach your next breakthrough. We are unlocking the future of technology.

From Hewlett-Packard to Agilent to Keysight.



www.keysight.com/find/nano

For more information on Keysight Technologies' products, applications or services, please contact your local Keysight office. The complete list is available at: www.keysight.com/find/contactus

Americas

Canada	(877) 894 4414
Brazil	55 11 3351 7010
Mexico	001 800 254 2440
United States	(800) 829 4444

Asia Pacific

Australia	1 800 629 485
China	800 810 0189
Hong Kong	800 938 693
India	1 800 11 2626
Japan	0120 (421) 345
Korea	080 769 0800
Malaysia	1 800 888 848
Singapore	1 800 375 8100
Taiwan	0800 047 866
Other AP Countries	(65) 6375 8100

Europe & Middle East

Austria	0800 001122
Belgium	0800 58580
Finland	0800 523252
France	0805 980333
Germany	0800 6270999
Ireland	1800 832700
Israel	1 809 343051
Italy	800 599100
Luxembourg	+32 800 58580
Netherlands	0800 0233200
Russia	8800 5009286
Spain	800 000154
Sweden	0200 882255
Switzerland	0800 805353
	Opt. 1 (DE)
	Opt. 2 (FR)
	Opt. 3 (IT)
United Kingdom	0800 0260637

For other unlisted countries:

www.keysight.com/find/contactus
(BP-9-7-17)