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High temperature nanoscratching of single crystal silicon under reduced oxygen condition

Saeed Zare Chavoshi[a], Santiago Corujeira Gallo[b], Hanshan Dong[b], Xichun Luo[c,*]

[a] Department of Mechanical Engineering, Imperial College London, Exhibition Road, London, SW7 2AZ UK
[b] School of Metallurgy and Materials, University of Birmingham, Edgbaston, Birmingham B15 2TT, UK
[c] Centre for Precision Manufacturing, Department of Design, Manufacture and Engineering Management, University of Strathclyde, Glasgow G1 1XJ, UK

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ABSTRACT

In-situ high temperature nanoscratching of Si(110) wafer under reduced oxygen condition was carried out for the first time using a Berkovich tip with a ramp load at low and high scratching speeds. Ex-situ Raman spectroscopy and AFM analysis were performed to characterize high pressure phase transformation, nanoscratch topography and nanoscratch hardness. No remnants of high pressure silicon phases were observed along all the nanoscratch residual tracks in high temperature nanoscratching, whereas in room temperature nanoscratching, phase transformation showed a significant dependence on the applied load and scratching speed i.e. the deformed volume inside the nanoscratch made at room temperature was comprised of Si-I, Si-XII and Si-III above different threshold loads at low and high scratching speeds. Further analysis through AFM measurements demonstrated that the scratch hardness and residual scratch morphologies i.e. scratch depth, scratch width and total pile-up heights are greatly affected by the wafer temperature and scratching speed.

1. Introduction

Silicon is a technologically crucial material and is the workhorse of the semiconductor industry due to its excellent stability, wear resistance and abundance. However, bulk wafers of single crystal silicon exhibit poor machinability at room temperature owing to their relatively low fracture toughness and high nanoindentation hardness. It is a common belief that the yield strength and hardness of silicon would reduce at high temperature. As such, the fracture toughness of silicon improves and its hardness decreases which would ease the plastic deformation and improve the machinability [1,2].

Molecular dynamics (MD) simulation studies have been conducted on high temperature nanometric cutting of silicon for the sake of making important contributions to our fundamental understanding of the occurring processes at the atomic scale at elevated temperatures [1,3–5]. It has been revealed that MD simulation is a robust numerical analysis tool in addressing a range of complex nanometric cutting problems that are otherwise difficult or impossible to understand using other methods. For example, the mechanics of high temperature nanometric cutting of silicon is influenced by a number of variables such as machine tool performance, cutting conditions, material properties, and cutting tool performance (material microstructure and physical geometry of the contact) and all these variables cannot be monitored online through experimental examination. However, these could suitably be studied using an advanced simulation based approach such as MD simulation. Although MD simulation offers a unique opportunity to explore the atomic level discrete processes of nanometric cutting/scratching of silicon under desired conditions, there exists some other phenomena which are impossible to be investigated using MD simulation, attributable to either the lack of a proper interatomic potential function or the excessive intricacy of the phenomenon. For instance, the available potential functions are not robust in describing and capturing all the structural phases of silicon; hence phase transformation mechanisms during nanometric cutting/scratching at room and elevated temperatures cannot be understood through MD simulation. Furthermore, the MD simulated depth of cut is only several nanometres, which is much smaller than the actual depth of cut (~several hundred nanometres to tens of micrometres). Consequently, high pressure phase transformation cannot be simulated under such condition. Therefore, experimental determination of the formation of polymorphs is required. It should be noted here that the material removal mechanism in scratching is similar to that in cutting/machining. Hence, it is possible to substitute the complicated cutting/machining with a relatively simple scratching so as to study material removal mechanism involved in cutting/machining [6].

In this paper, the focus will be on the experimental studies of the

* Corresponding author.
E-mail addresses: s.zare@imperial.ac.uk (S.Z. Chavoshi), xichun.luo@strath.ac.uk (X. Luo).

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pressure-induced silicon polymorphs, nanoscratch topography and nanoscratch hardness in nanoscratching of silicon at room and elevated temperatures. To this end, using the state-of-the-art nanoindentation equipment, in-situ high temperature nanoscratching trials on single crystal silicon wafer under inert gas are performed and ex-situ characterization techniques, such as Raman spectroscopy and atomic force microscopy (AFM) are employed to shed some light on the aforementioned processes.

2. Literature review

The previous work on nanoscale cutting/scratching/indentation by MD simulation has primarily focused on demystifying the material removal mechanisms at “room temperature”. What is known from these studies is that the current pool of knowledge on the nanometric cutting/scratching/indentation at elevated temperatures is still sparse. Only limited work has been done so far on studying hot nanoscale cutting/scratching/indentation by MD. In preliminary investigations performed by the authors, hot nanometric cutting of single crystal silicon and silicon carbide on different crystal orientations was compared with the cutting at room temperature (27 °C) so as to characterize the cutting mechanics such as material flow, stagnation region, specific cutting energy, cleavage and defect-mediated plasticity [1,3–5,7–9]. Fang et al. [10] and Liu et al. [11] performed MD simulations to examine the variation in Young's modulus, hardness and elastic recovery of copper, diamond and gold during nanoindentation at high temperatures (up to 327 °C). They concluded that Young's modulus, hardness and the extent of elastic recovery decreases with the increase of temperature. Hsieh et al. [12] used MD simulation to investigate the effect of temperature on maximal normal forces and elastic recovery during nanoindentation of copper. They reported a reduction in the aforementioned parameters with an increase in the substrate temperature.

On the experimental side, there is no study hitherto on high temperature nanometric cutting/scratching. However, there is a history of using ‘hot hardness’ microindentation tests, beginning with Atkins and Tabor [13]. Nevertheless, instrumented hardness tests at high temperatures have a shorter history. Wheeler and co-workers [14–16] have described the general nano-mechanical test platform capable of performing variable temperature and variable strain rate testing. The thermal management and measurement techniques and vacuum nanoindentation have been discussed in their review papers. Similarly, Schuh and his colleagues [17,18] produced an elegant discussion of the technical issues surrounding high temperature nanoindentation in ambient and inert environments.

In 1996, Suzuki and Ohmura [19] performed ultra-microindentations on {110} surfaces of single crystal silicon in the temperature range of 20–600 °C and concluded that below 500 °C, the temperature-insensitive hardness is determined by the transformation to the metallic β-tin phase, which amorphizes or nanocrystallizes during unloading, while above 500 °C, plastic deformation due to dislocation activity causes temperature-dependent hardness. Smith and Zheng [20] modified a depth sensing indentation instrument to measure small scale hardness and elastic modulus of glass, gold, and single crystal silicon at 200 °C. The hardness and elastic modulus of soda lime glass and gold were found to be lower than that at room temperature. In contrast, indentation testing of Si(100) at 200 °C produced a similar hardness value to that obtained at room temperature, although the modulus was reduced, from 140.3 to 66 GPa. In addition, the well-known ‘pop out’ event, which is observed during unloading of a silicon indentation at room temperature, disappeared at 200 °C. Beake and Smith [21] demonstrated that mechanical properties of fused silica exhibit a completely different temperature dependence from those of soda-lime glass during high temperature nanoindenation at 400 °C, since fused silica is an anomalous glass. Xia et al.[22] observed that the surface hardness of Fe-40Al, an iron aluminide, is higher and the elastic modulus is lower at elevated temperatures (400 °C) than the corresponding values at room temperature. Lund et al.[23] investigated the effect of temperature during nanoindentation of pure platinum. They reported that the transition from elastic to plastic deformation takes place at progressively lower stress levels as temperature is increased. By adapting a commercial nanoindenter to allow testing at up to 200 °C, Schuh et al.[24] explored the deformation map of two type of metallic glasses, and found that increasing the temperature at a constant indentation rate sees the gradual emergence of homogeneous flow, as thermal relaxations allow dissipation of strain localization into general viscous flow. Nanoindentation studies of single crystal Ni-base superalloy CMSX-4 oriented in the $<001>$ and $<110>$ directions were conducted by Sawant and Tin [25] over a range of temperatures from 30 °C to 400 °C. Trelewicz and Schuh [26] carried out high-temperature nanoindentation experiments to assess the activation enthalpy for deformation of nanocrystalline Ni-W alloys, for grain sizes between 3 and 80 nm. They reported that thermal softening becomes less pronounced at finer grain sizes, and the activation enthalpy has an apparent inflection at a grain size near ~10–20 nm, in the vicinity of the Hall-Petch breakdown. It should be noted here that large amount of studies have been performed on the high temperature nanoindentation of various materials [27–36]. However, for the sake of brevity, only studies on silicon are discussed in the following paragraphs. Bradby and his co-workers [37,38] reported that in hot nanoindentation of silicon, increasing temperature enhances the nucleation of Si-III and Si-XII during unloading but the final composition of the phase transformed zone is also dependent on the thermal stability of the phases in their respective matrices. Besides, they found that the region under the indenter undergoes rapid volume expansion at temperatures above 125 °C during unloading. Moreover, polycrystalline Si-I was the predominant end phase for indentation in crystalline silicon whereas high-pressure Si-III/Si-XII phases were the result of indentation in amorphous silicon. They also concluded that the Si-II phase is unstable in a c-Si matrix at elevated temperatures. In a similar work, Domnich et al.[39] carried out high-temperature nanoindentation using Berkovich probe and observed that up to a certain critical temperature (350 °C), the nanoindentation hardness of silicon is dictated by the pressure required to transform the semiconducting Si-I phase into the metallic Si-II phase of silicon. However, no phase transformation was observed above 350 °C and it was suggested that the nanoindentation hardness in silicon above 350 °C is dictated by dislocation glide. From what was discussed above, it can be inferred that although some studies have been performed so as to improve our understanding of high temperature nanoindentation behaviour, no methodical work is available to date on the area of high temperature nanometric cutting/scratching of silicon. It might be argued that both techniques are beneficial in understanding and characterizing the materials; nevertheless, nanometric cutting/scratching unlike nanoindentation is dominated by deviatoric stresses carrying pronounced component of shear. Consequently, the stress distribution in nanometric cutting/scratching is considerably different from that of nanoindentation; hence the results are not transferable. Accordingly, it is suggested that there is a strong need to understand the high temperature nanometric cutting/scratching mechanisms of hard-brittle materials such as silicon.

3. Experimental setup and test procedure

3.1. Equipment

Nanoscratching trials were performed on a MicroMaterials Ltd. (MML) nanoindenter called NanoTest Vantage. This equipment permits testing at elevated temperatures with low thermal drifts under reduced oxygen/purged condition and controlled humidity levels, which offers the perfect capability for testing materials in extreme conditions. Fig. 1 demonstrates the heating arrangement in the MML