

**COMMENT**Worldwide Congress on
Materials and Manufacturing
Engineering and Technology16th - 19th May 2005
Gliwice-Wiśła, PolandCOMMITTEE OF MATERIALS SCIENCE OF THE POLISH ACADEMY OF SCIENCES, KATOWICE, POLAND
INSTITUTE OF ENGINEERING MATERIALS AND BIOMATERIALS OF THE SILESIA UNIVERSITY
OF TECHNOLOGY, GLIWICE, POLAND
ASSOCIATION OF THE ALUMNI OF THE SILESIA UNIVERSITY OF TECHNOLOGY, MATERIALS
ENGINEERING CIRCLE, GLIWICE, POLAND**13th INTERNATIONAL SCIENTIFIC CONFERENCE
ON ACHIEVEMENTS IN MECHANICAL AND MATERIALS ENGINEERING**

Implications of scale effects on the mechanical response

Y.Katz and W.W Gerberich

University of Minnesota, MN, 55450, U.S.A

Abstract: In elastic-plastic solids by approaching the sub micron scale, critical experiments have confirmed major differences in the mechanical response. In small volume segments, the major difference is manifested in increasing values of the hardness or strength. Specifically, the study is centered on ultra fine silicon particles in the range of 20-50 nm on sapphire substrate. Mechanical tests have been performed by contact mechanics methodology at ambient temperature. The mechanical and visualization information have been achieved by novel techniques assisted by scanning probe microscope-based nano indentation. At this stage, the findings alluded to a model that has been founded on dislocation dynamic effects. Beside evaluations, regarding fatigue and structural integrity aspects, it became apparent that even macro events might gain better assessment due to the role of localization confined by definition to an extremely small volume situation.

Keywords: Silicon, Contact mechanics, Nano scale, Reverse plasticity.

1. INTRODUCTION

Critical experiments have substantiated already the role of mechanical the size effect. Such experiments in various metallic systems included nano indentation findings, wire torsion and fine sheet bending in terms of hardness, strengths and plasticity behavior [1-3]. In fact, the major scale effect as related to mechanical properties is manifested by the increase values of strengths or hardness as the volume decreases. The mechanical response of small volume segments provides enough incentives for continuous research activities on different levels. Briefly, at least two models have been proposed regarding the length scale effect. First, Hutchinson [3] has developed the generalization of the classical plasticity theory. As such, the length scale has been introduced to account for plastic strain gradient effects emerging in deformation at sub micron scale. Following this approach, size effects are attributed to the increasing preponderance of geometry necessary dislocation relative to statistically stored dislocation. This is extenuated in cases in which the scale of the formation decreases. Second, mainly under indentation the surface to volume ratio approach introduces a length scale by considering this ratio to become a key parameter [4]. The current investigation remains phenomenological and is centered on the mechanical response of ultra fine silicon particles tested under monotonic and cyclic loading at ambient temperature. Beside the implications to ultra fine particles or thin films such activity might facilitate better understanding even regarding macro events. Due to localization, fracture and fatigue processes are typical examples as related to this category.

2. MATERIALS AND EXPERIMENTAL PROCEDURES

Identified nano spheres of silicon particles in the range of 20-50 nm were selected. The nano particles were synthesized by injecting vapor phase of silicon tetrachloride into argon-hydrogen thermal plasma. The material in this plasma state was then expanded through a nozzle to a low pressure, a process that drove the nucleation of silicon nano particles. This synthesis known as hypersonic plasma deposition was then utilized in a focused beam and the nano particles were directed across a sapphire wafer (Fig.1). The substrate was mounted on a computer-controlled translation system and allowed the deposition of nano particles lines. At this stage particles could be accessed with the aid of scanning probe microscope based nano indenter. Here, Hysitron Triboscope, Hysitron Inc, Edina MN, USA, was utilized. For remote loading, a diamond of 1000nm tip radius was used at ambient temperature. In fact, the experimental conditions were a diamond indenter with elastic modulus of 1100GPa on one side and Al_2O_3 single crystal substrate with 450GPa modulus on the other. Using this set-up implies that the silicon nano particles with a defined geometry was compressed between two relatively rigid platens. The ultra fine particles were also confirmed by selected area diffraction assisted by Philips CM30 Transmission Electron Microscope (TEM) operating at 300KV (Fig.2). In fact, under the current circumstances of contact stresses the initial deformation was analyzed by contact mechanics methodology.

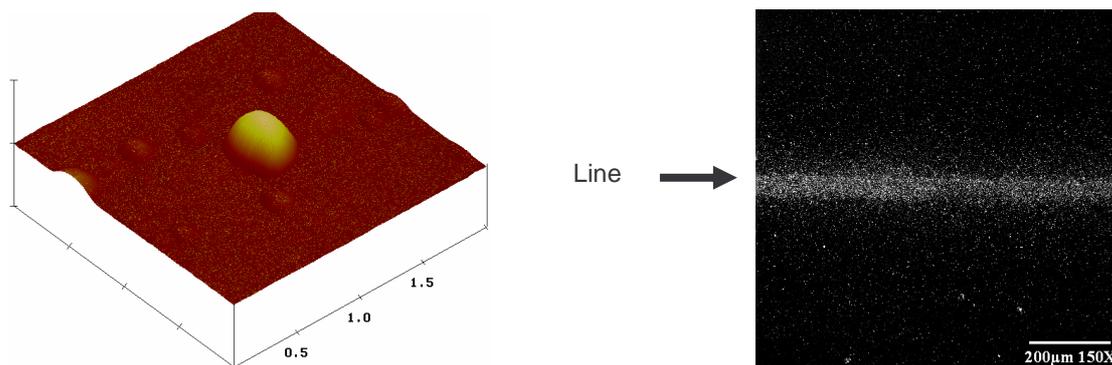


Figure1. (Left) Individual particle (Right) Line deposition of Si single crystals

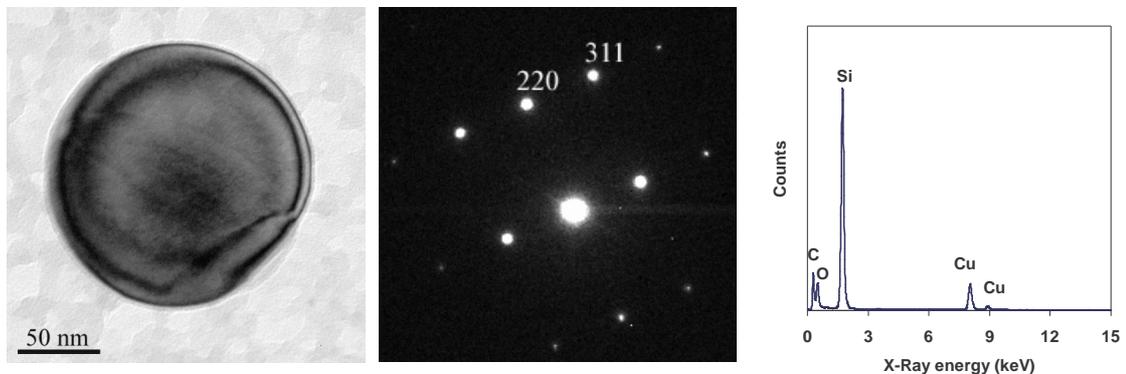


Figure 2. (Left) X-ray analysis of particle confirms it is Si. (Center) Selected area diffraction pattern of this particle zone axis of Si (Right) TEM of typical spherical Si particle, 80 nm radius

3. EXPERIMENTAL RESULTS

In nano particles of Si with modulus of 170GPa at ambient temperature the scale effect became apparent. Mainly it has been manifested in a consistent trend that the hardness or strength is size dependent. Smaller size resulted in higher deformation resistance. For example, the hardness of silicon nano spheres has indicated a factor of four higher values as compared to <100> bulk silicon. This behavior prevailed in testing nano spheres of various dimensions. In the case of a particle of about 19nm the hardness exceeded by far the aforementioned factor for a specific load history. With indentations monotonic or cyclic, information as related to geometry, displacement and the recovery could be achieved. Particularly in fatigue argumentations the role of the irreversible strain or the cumulative value of damage could be measured. This loading history is clearly a low cycle fatigue experiment even by considering the frequency conditions of 0.3 Hz. In spite such limitations, important insights have been revealed regarding plasticity of silicon at ambient temperature in general and mechanical aspects has related to small volume in particular.

4. DISCUSSION

Beside the significant hardness increase by almost a factor of five nano silicon particles as compared to bulk value, additional striking results have been noticed. Under the present circumstances of contact stresses, plasticity resulted with substantial strain hardening pattern. Such behavior by it self is alluding to possible susceptibility to cyclic loading. The role of cumulative damage was experimentally confirmed. Kahn et al [5] and Komai et al [6, 7] have addressed the pre mature failure by fatigue in silicon based films. Another finding requires additional attention. Silicon has been accepted as a brittle system with highly hard Ductile-Brittle transition behavior that only occurs at about 773K. This means that at lower temperatures dislocation activity is not accepted. Moreover, under cyclic load by repeated run of indentations, the present super hard nano particles of silicon, indicated changes in the hysteresis pattern. In this context, the displacement consisted in a partition nature of reverse and irreversible plastic strain. This partition that could be approached quantitatively actually indicated that plastic behavior was size dependent. In some cases the load-displacement curve enabled to track a staircase yield excursions attributed to dislocations injection. The origin mechanism that might cause hardening as well as cumulative damage becomes apparent. Here to mention the argument, that in brittle material, fatigue occurs by cycle dependent degradation of the extrinsic toughness in the wake of the crack tip. Accordingly, cyclic loading or friction wear degradation is attributed to the reduction of the extrinsic crack tip shielding [8, 9]. Nevertheless, under contact mechanical methodology dislocation mobility occurs and the argumentation of native oxide initiation sites or dynamic environmentally enhanced fracture is not decisive. Contact test under load controlled conditions requires special attention to the remote applied stress values. Variation of the contact area modifies the average stress and changes the applied stress amplitude. Kobayashi et al [10] investigated deformation of Al₂O₃ single crystals under repeated indentation. The study concluded that analogy of the sapphire response to the conventional S-N fatigue curves in metallic system can be deducted. Even in ceramic materials, plasticity occurs and above some thresholds twinning has been observed with crystallographic habits. Thus, in addition to well established view regarding the thermal role in silicon, even at ambient temperatures semi brittle behavior resulted. This means, that a more comprehensive view must include also the role of the state

of load. The aforementioned issue remains important not only regarding the scale factor but also to structural integrity aspects of small volume systems.

5. CONCLUSION

1. Silicon nano spheres in the 20-50nm radii range the hardness increased to 50 GPa as compared to values of about 10GPa for silicon bulk.
2. Super hard behavior under indentation beside other procedures raises the issue of the length scales as related to mechanical response.
3. At ambient temperatures a semi brittle behavior occur indicating work hardening and thus, cyclic damage susceptibility.
4. Dislocation activity model can provide clarification to the experimental findings.

ACKNOWLEDGMENTS

Experimentally based findings and nano particles processing developments by W.Mook, R.Mukherjee, A.Gidwani, J.Deneen at the Dep. of Chemical Eng. and Materials Science and the Dep. of Mech. Eng. - University of Minnesota are acknowledged and highly appreciated.

REFERENCES

1. Q.Ma.and D.R.Clarke; *J.Mater.Res.* Vol 10,p.853,1995.
2. W.D.Nix and H.Gao; *J.Mech.Phys.Solids.* Vol 46,p.411.1998.
3. J.W. Hutchinson; *Materials Science for the 21st century*, Vol A, JSMS. p.307,2001.
4. W.W.Gerberich, N.I.Tymiak, J.C.Grunlan. M.F.Horstemeyer and M.I.Baskes; *J of App Mech.* Vol 69,p.433,2002.
5. H.Kahn, R.Balarini, R.L.Mullen and A.H.Heuer; *Proc.Roy.Soc.A*, Vol 455,p.3807,1999.
6. K.Komai, K.Minoshima and S.Inoue; *Micros.Tech.* Vol.5,p.30,.1998
7. K.Komai, K.Minoshima and T.Terada; *The 36th strength design safety evaluation meeting*, *Soc.Met.Sci.Japan.* Vol 83,2000.
8. R.O,Ritchie; *Int.J.Frac.* Vol 100,p.55,1999.
9. C.L.Muhlstein. E.A.Stach and R.O,Ritchie; *J.Engng.Integ.Soc.* Vol 14,p.4,2003.
10. S.Kobayashi, Y.Uesugi and S.Miura; *J.Soc.Mat.Sci.Japan.* Vol 52,p.1166,2003.