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Further considerations on the high-cycle fatigue of micron-scale polycrystalline silicon

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Abstract—Bulk silicon is not susceptible to high-cycle fatigue but micron-scale silicon films are. Using polysilicon resonators to determine stress-lifetime fatigue behavior in several environments, oxide layers are found to show up to four-fold thickening after cycling, which is not seen after monotonic loading or after cycling in vacuo. We believe that the mechanism of thin-film silicon fatigue is "reaction-layer fatigue", involving cyclic stress-induced thickening of the oxide and moisture-assisted cracking within this layer.
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Keywords: MEMS; Silicon; Fatigue; Reaction-layer fatigue

Although the first report of fatigue of micron-scale silicon was published over 15 years ago [1] and there have been numerous studies since (recently reviewed in Refs. [2,3]), there is still no consensus on the fundamental mechanisms that cause this phenomenon. The problem is not only important technologically, i.e. for the reliability and durability of silicon microelectromechanical systems (MEMS), but also because it presents a fascinating scientific problem in that cyclic fatigue failures do not occur in macroscale silicon[1] whereas they do at the micron scale (in both mono- and polycrystalline material).

Classically, fatigue cracking under tension/compressive or bending is associated with crack-tip blunting/sharpening due to cyclic plasticity in ductile materials, and with the degradation of extrinsic toughening (crack-tip shielding) mechanisms in the wake of the crack tip in brittle materials [7]. In the absence of plasticity, as in the latter case, the mechanism of crack advance remains essentially unchanged from that under monotonic loads; however, cyclic loading degrades the shielding mechanisms, such as crack bridging, in the crack wake, which in turn leads to a larger crack-driving force at the crack tip. As no such phenomena have been observed in silicon at ambient temperatures, the occurrence of the fatigue of micron-scale silicon films is at first sight somewhat of a mystery. In the current literature [2,3], however, two main classes of explanations have been advanced, namely mechanisms that assume subcritical crack growth and/or mechanical damage in the silicon itself [2,8,9], and those that involve the cyclic-induced thickening and subsequent moisture-induced subcritical cracking of the surface oxide layer [10–14].

Our work has focused largely on traditional stress-lifetime (S/N) fatigue experiments of polycrystalline silicon (polysilicon) MEMS comb-drive actuated fatigue resonators (Fig. 1), which are cycled at a constant displacement (with a load ratio[2] of -1) up to the point of failure [11,14]. Capacitive sensing of the displacement

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[1] Silicon has been reported to exhibit "fatigue" under repetitive indentation loading [4–6], as noted below, although we believe that this is an entirely distinct phenomenon of questionable relevance to the fatigue cracking of thin films of silicon in bending or tension/compression loading conditions.

[2] The load ratio R is defined as the ratio of the minimum to maximum loads in the fatigue cycle.

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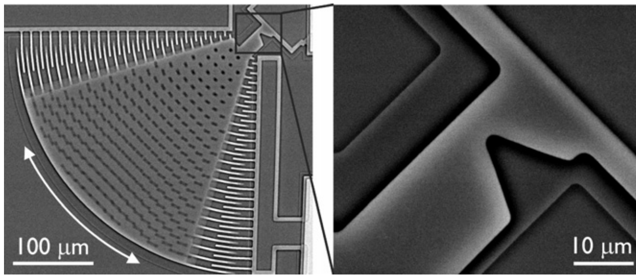

Figure 1. Scanning electron micrographs of the polysilicon MEMS fatigue resonator. There is a triangular free-standing proof mass with an interdigited comb drive on one side and capacitive displacement sensor combs on the other side in motion in situ in the SEM ($R = -1$; $f_{res} \sim 40$ kHz); in the image the edges of the comb fingers are blurred because of the high-frequency motion. The blow-up shows the notched cantilever beam connecting the resonator mass to the anchor.

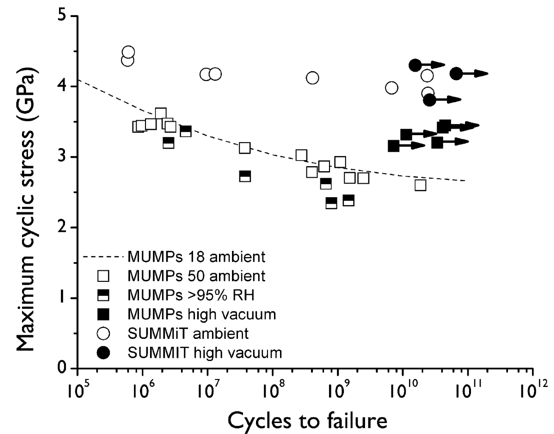

Figure 2. Combined maximum cyclic stress-lifetime (S/N) data (at $R = -1$) for polysilicon MUMPs and SUMMiT devices. The devices were tested in ambient air (30–40% RH), in high relative humidity air (>95% RH) and under very high vacuum ($<2.0 \times 10^{-7}$ mbar). The arrows for the vacuum data points represent run-outs, i.e. tests that were stopped after no failure occurred.

is measured throughout the test such that the structure remains under constant stress amplitude loading by periodically updating the resonance frequency. Moreover, by monitoring the change in resonance frequency, the accumulation of damage can also be evaluated during the test [11,14]. The ability to monitor the degradation of the silicon film prior to failure provides insights into the fatigue mechanisms that cannot be gleaned from failure data alone. We have collected S/N data at ~25 °C in ambient air, both at 30–40% relative humidity (RH) and in high vacuum ($<2.0 \times 10^{-7}$ mbar) for devices fabricated in both the PolyMUMPs^{TM} (MUMPs)[3] and Sandia SUMMiT V^{TM} (SUMMiT) processes.[4] Additionally, MUMPs specimens were run in ambient air at >95% RH [14]. Transmission electron microscopy (TEM) was then used to examine devices that were either fatigued, cycled in vacuo or fractured monotonically using a probe tip (i.e. without cycling). The MUMPs devices (Si film thickness ~2 μm) were observed using the Berkeley JEOL high voltage atomic resolution microscope at 800 keV in bright field, which mitigates the need for TEM sample preparation. The SUMMiT devices (Si film thickness 2.5 μm) were imaged using energy-filtered TEM (EFTEM) in a Philips CM200FEG at 200 kV equipped with a Gatan image filter after the samples were prepared for TEM imaging by focused ion beam [14].

Our results in Figure 2 show that micron-scale polysilicon displays classical S/N fatigue behavior with progressively decreasing lifetimes with increasing applied cyclic stresses for all samples tested in air. However, fatigue lifetimes at a given applied stress were shorter in a high-humidity environment and no fatigue failures at all were observed in high vacuum, even with high maximum stresses of 3–4.5 GPa for in excess of 10^{10} cycles. A decay in the resonance frequency was found to accompany the tests run in air [11,14], suggesting stable damage accumulation up to failure, whereas no such resonant frequency changes were seen for tests in vacuo [14], consistent with a lack of any damage accumulation.

It is hard to believe from these results (and from conventional mechanistic notions of fatigue) that the fatigue failure of thin-film polysilicon can be a purely mechanical effect. Clearly, polysilicon devices with both thick (~20 nm) and thin (~4 nm) initial (post-release) oxide layers display S/N fatigue behavior that is markedly affected by the environment. Indeed, our results are consistent with published studies on micron-scale silicon that show faster damage accumulation in higher humidity environments [15] and a slower damage accumulation rate (or "absence of failure") when the service environment or surfaces of the film are modified [8]. TEM characterization of MUMPs and SUMMiT devices fatigued at $\sim 10^{10}$ cycles (Fig. 3) confirm our initial observations (first published in Ref. [10]) that cycling in air leads to a thickening, in this case by a factor of approximately four, of the oxide layer in the higher stressed region at the notch root (Fig. 3A, C and D). No such oxide thickening was observed after cycling for the same number of cycles in vacuo (Fig. 3B, E and F), nor for fracture under monotonic (non-cyclic) loads. Moreover, high-voltage TEM imaging of fatigued MUMPs devices showed not only such thickened oxide layers after cyclic loading but also the presence of subcritical cracks, tens of nanometers in length, within the oxide layer of devices that had been stopped prior to failure [10,11].

Silicon is known to be immune to environmentally induced cracking, whereas silica is highly susceptible [16]. Consequently, our results led us to propose a "reaction-layer fatigue" mechanism for high-cycle fatigue of thin-film silicon [10–14], where the fatigue process occurs not in the silicon itself, but by moisture-induced cracking in the cyclic stress-assisted thickened oxide layer. Provided the oxide layer is thick enough, stress and moisture-assisted cracking in the oxide can create a crack large enough to break the entire device, i.e. the oxide cracks become large enough to exceed the critical crack size

[3] Fatigue tests were run on the MUMPs devices from two fabrication runs (nos. 18 and 50).

[4] Two different fabrication sources for the polysilicon devices were chosen as the MUMPs devices have a much thicker post-release oxide layer (~20 nm) than the SUMMiT devices (~4 nm).

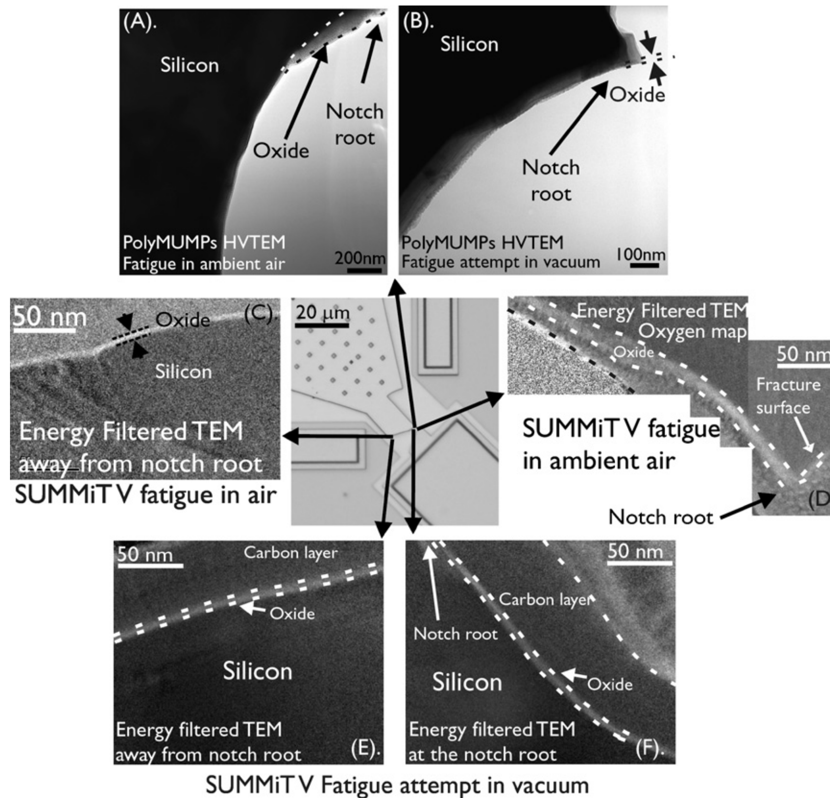

Figure 3. TEM images showing the silicon oxide thickness of the MUMPs and SUMMiT devices at a stress concentration after testing: (A) HVTEM of the notch section of MUMPs fatigued in ambient air (30–40% RH, ~100 nm oxide at the notch, ~20 nm oxide away from the notch); (B) HVTEM of the notch section of the MUMPs fatigue attempt in high vacuum (<2×10^{-7} mbar, ~20 nm oxide); (C) EFTEM away from the notch of SUMMiT fatigued in air (~4 nm); (D) EFTEM at the notch of SUMMiT fatigued in air (~15 nm oxide); (E) EFTEM away from the notch of the SUMMiT fatigue attempt in vacuum (~5 nm); and (F) EFTEM at the notch of the SUMMiT fatigue attempt in high vacuum (~5 nm).

for the entire structure. Reaction-layer fatigue thus requires the critical crack size to be comparable to the oxide thickness; since this would never occur in a macroscopic component of silicon, this mechanism provides an explanation of why silicon fatigue is confined to micron-scale components [17,18]. Fracture mechanics calculations show that critical crack sizes are on the order of 50 nm if the crack reaches critical size before reaching the silicon/silicon oxide interface [17] but can be as low as 15 nm or smaller when instability occurs at the interface [18]; these numbers are consistent with the oxide thicknesses we observed at the notch root (Fig. 3). However, the details of the relationship between the evolution of the surface oxide and the growth of the crack have remained elusive. Finite-element models of the oxide–silicon bimaterial system have provided important insight into the range of oxide thicknesses, peak stresses and crack-tip locations that are relevant to reaction-layer fatigue [17,18]. Unfortunately, the idealizations in the model (e.g. smooth side walls) and the inability to capture the cycle-dependent evolution of the system make it difficult to accurately compare the fatigue resistance of various silicon films. Although the precise physical mechanisms associated with the cyclic-stress-assisted thickening of the oxide layer are not fully understood, we know that the process is not thermal, as our in situ high-resolution IR imaging of the notch root showed no significant rise in temperature there [11]. Several researchers have independently confirmed the presence of thickened oxides during thin-film silicon fatigue [12,19]. For example, atomic force microscopy imaging by Allameh et al. [12] of polysilicon resonator samples, similar to those shown in Figure 1, revealed a surface roughening effect at stress concentrations during cyclic loading; these authors suggested a complementary fatigue mechanism involving stress-assisted oxide thickening, caused by dissolution of the surface oxide, thereby forming deep grooves in the vicinity of the notch that become sites for crack initiation.

Although all our results, and those of many other authors (see Ref. [3]), are completely consistent with the notion of reaction-layer fatigue, as pointed out by Heuer and co-workers [20], there are certain results by other researchers which may be construed as being inconsistent with this mechanism. One of these is that S/N fatigue lifetimes for thin-film silicon appear to be frequency-independent [19,21], i.e. lifetimes depend on the number of cycles and not on time. At first, this observation would seem to be inconsistent with any environmentally induced mechanism. However, the process of moisture-induced cracking in the oxide layer is certainly not conventional stress-corrosion cracking as first proposed by Connolly and Brown [1], as it initially involves the process of the thickening of the oxide. Although the rate-limiting step involved in reaction-layer fatigue is not known, we do know that the oxide thickening process is not time-dependent (it does not occur under monotonic loading); rather, it is a function of

the fatigue cycling. Measurements of the notch-root oxide thickness as a function of the number of fatigue cycles would certainly help clarify this issue, although such experiments would be extremely difficult and tedious to perform.

A second, and related, issue raised by Heuer and co-workers [20] is the absence of a fatigue effect under static (non-cyclic) loading [8,20]. Whereas this effect is certainly true, it is not inconsistent with a reaction-layer mechanism as the one integral part of the fatigue process, that of oxide thickening, only occurs under cyclic loading. In the absence of oxide thickening, cracks in the oxide layer may never get large enough to cause complete failure of the device.

A more important issue is the strong dependency of load ratio on the fatigue behavior of polysilicon which has been observed by Heuer and co-workers [9,20]. Specifically, they report a decrease or increase in the fracture strength (measured under monotonic loading) following prior fatigue cycling at different load ratios, with the extent of apparent weakening or strengthening depending upon the value of load ratio. Devices that have been pre-cycled at a high compressive or tensile mean stress with a small cyclic stress appear to have a higher subsequent fracture strength, whereas those cycled at a low mean stresses with a high cyclic stress have a lower fracture strength, all compared to the fracture strength of the uncycled device [9]. These results have recently been confirmed by Boroch and Bagdahn [22]. It is pertinent to note here that these experiments are inherently different from the S/N fatigue tests presented in Figure 2, where a constant cyclic stress is applied up to the point of catastrophic failure. Having said that, whereas a weakening effect due to prior cyclic loading is certainly nominally consistent with the damage associated with reaction-layer fatigue, we are at a loss to explain the corresponding strengthening effect at high positive or negative R ratios. Heuer et al. [8,9,20] suggest that these results imply that thin-film silicon fatigue involves a mechanical component based on subcritical damage accumulation in the silicon itself, although they present no viable mechanisms for this effect. Their proposed mechanism involving grain-boundary plasticity, although theoretically feasible for nanocrystalline silicon [23], cannot account for similar observations of thin-film fatigue in grain boundary-free (i.e. single crystal) silicon [1,3,15,24]. While it is possible that the large compressive stresses associated with the high load ratios introduce an additional source of fatigue damage, we believe that the strengthening/weakening behavior of polycrystalline silicon films does not invalidate the reaction-layer fatigue mechanism.

Apparent support for the notion that Si fatigue results from mechanical damage accumulated in the silicon itself has recently been presented by Bhowmick et al. [4], who claim there exists a mechanical fatigue effect in bulk silicon. However, as these experiments were based on repetitive macroscale indentation tests under extremely high compressive loads, we do not believe that they pertain to the process of fatigue-crack growth in bulk or especially thin-film silicon. For a start, the indentation loading geometry is inherently different from bending/tensile testing loading conditions and the applied loads are far higher. Moreover, (cyclic) indentation in silicon was the subject of several comprehensive studies [5,6,25,26] prior to Bhowmick et al.'s work [4], and several different deformation, fracture and phase transformation effects have been reported for both bulk and micron-scale silicon under the influence of high compressive and shear loads during indentation, which we believe have little relevance to thin-film silicon bending fatigue. These effects include in situ phase transformations to an amorphous, R8/BC8 or Si-XII crystal structure [5,6,25], the presence of radial/median cracks [5,6] and the emission of dislocations at room temperature [5,6,25,26].

To amplify this latter effect, we note that, in all cases during the indentation of silicon, high residual densities of defects are observed in the vicinity of the indentation. This is because, upon the removal of the applied load, there are insufficient stresses to permit further motion of the dislocations at room temperature – in effect, the dislocation array is frozen in place. This is an important point; only during the application of high compressive and shear loads are there stresses sufficiently high to nucleate dislocations and propagate them into the sample bulk. When the load is removed, there is insufficient thermal energy to allow the dislocation array to experience any dynamic recovery, i.e. there is no possibility of the thermally induced dislocation kink nucleation and propagation necessary for dislocation motion [27,28]. Thus, these indentation studies indicate that if dislocations were to be formed during fatigue loading, one would expect to observe them in the microstructure after failure. To date, there have been absolutely no reports of such observations in any of the relevant thin-film silicon literature [3,9–11,13,14,20].

The external surfaces of silicon films and their interaction with fabrication and service environments have been directly correlated with fatigue. For example, surface treatments with a hydrophobic (organic) monolayer [11], the desorption/adsorption of water via thermal and pressure cycling or changes in relative humidity [15], and the enhanced oxidation of the silicon (e.g. via galvanic effects) have all been shown to cause marked changes in damage accumulation rate and life. In all of these cases, the underlying mechanisms have been linked to localized, cycle-assisted oxidation and crack growth in the said layer [29]. Even in the extreme case of the oxide-free silicon created by the deposition of a hydrophobic nanoscale coating, fatigue failure was accompanied by localized oxidation and cracking of the film surfaces [10,11,15,29]. Significant confusion has arisen over the apparent differences in fatigue susceptibility and surface condition after cyclic loading [8,20]. As demonstrated above, the processing history and testing conditions can have a marked effect on fatigue life. Consequently, testing methods that require the application of conductive coatings to specimens prior to testing [8,20] are likely to confound the testing environment and surface condition. Given the sensitivity of fatigue degradation to the surface condition, it is not surprising that tests conducted on Pd-coated surfaces might be influenced by the noble, conductive layer. Specifically, localized oxidation and cracking would be restricted to regions where the film was discontinuous or compro-

mised by the cyclic loading, and would be extremely difficult to document with TEM. Such a configuration would also have a significant effect on the adsorption of water to the surface, and lead to the apparent discrepancy between behavior of silicon films in a medium vacuum [8,15]. Consequently, we feel that one should exercise caution before dismissing the importance of cyclic oxidation and moisture on fatigue-crack growth in silicon films.

We conclude that the evidence for a significant environmental contribution to the fatigue of micron-scale silicon films is irrefutable, as shown principally by our observations that fatigue lifetimes are shorter in high-humidity atmospheres and that Si films tested in a high vacuum do not suffer delayed fatigue failures. Further, based on our TEM studies, we believe that the principal mechanism underlying this behavior is reaction-layer fatigue [10,11], involving the cycle-induced thickening of the oxide layer at the notch root and moisture-induced subcritical cracking within this layer. The open question that remains is whether there is an additional mechanical fatigue effect involving cyclic damage in the silicon itself. Although it is hard to conceive mechanistically what such progressive cyclic damage could be in a prototypical brittle material such as silicon – in fact, no viable mechanisms have been proposed to date – this does remain a possibility in light of results showing strengthening after prior fatigue cycling at high R values and the occurrence of delayed fatigue failures in Pd-coated polysilicon. Clearly more research will be required to resolve this issue. Indeed, as noted by Kahn et al. [20], “The mechanism(s) responsible for silicon fatigue will remain a disputed topic until a comprehensive explanation is developed”.

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References

[1] J.A. Connally, S.B. Brown, Science 256 (1992) 1537.
[2] H. Kahn, R. Ballarini, A.H. Heuer, Current Opinion in Solid State & Materials Science 8 (2004) 71.
[3] D.H. Alsem, O.N. Pierron, E.A. Stach, C.L. Muhlstein, R.O. Ritchie, Advanced Engineering Materials 9 (2007) 15–30.
[4] S. Bhowmick, J.J. Melendez-Martinez, B.R. Lawn, Applied Physics Letters 91 (2007) 667–678.
[5] I. Zharudi, L.C. Zhang, M.V. Swain, Journal of Materials Research 18 (2003) 758–761.
[6] I. Zharudi, L.C. Zhang, M.V. Swain, Proceedings of the Institution of Mechanical Engineers Part C – Journal of Mechanical Engineering Science 218 (2004) 591–598.
[7] R.O. Ritchie, International Journal of Fracture 100 (1999) 55.
[8] H. Kahn, R. Ballarini, J.J. Bellante, A.H. Heuer, Science 298 (2002) 1215–1218.
[9] H. Kahn, L. Chen, R. Ballerini, A.H. Heuer, Acta Materialia 54 (2006) 667.
[10] C.L. Muhlstein, E.A. Stach, R.O. Ritchie, Applied Physics Letters 80 (2002) 1532.
[11] C.L. Muhlstein, E.A. Stach, R.O. Ritchie, Acta Materialia 50 (2002) 3579–3595.
[12] S. Allameh, P. Shrotriya, A. Butterwick, S. Brown, W.O. Soboyejo, Journal of Microelectromechanical Systems 12 (2003) 313.
[13] D.H. Alsem, E.A. Stach, C.L. Muhlstein, R.O. Ritchie, Applied Physics Letters 86 (2005) 041914-1.
[14] D.H. Alsem, R. Timmerman, B.L. Boyce, E.A. Stach, J.T.M. De Hosson, R.O. Ritchie, Journal of Applied Physics 101 (2007) 013515-1.
[15] O. Pierron, C.L. Muhlstein, Journal of Microelectromechanical Systems 15 (2006) 111.
[16] S.M. Wiederhorn, Journal of the American Ceramic Society 55 (1972).
[17] C.L. Muhlstein, R.O. Ritchie, International Journal of Fracture 120 (2003) 449.
[18] O.N. Pierron, C.L. Muhlstein, International Journal of Fracture 35 (2005) 1.
[19] J. Bagdahn, W.N. Sharpe, Sensors and Actuators A 103 (2003) 9.
[20] H. Kahn, A. Avishai, R. Ballarini, A.H. Heuer, Scripta Materialia 59 (2008) 912–915.
[21] T. Namazu, Y. Isono, in: 17th IEEE International Conference on Micro Electro Mechanical Systems (MEMS): Maastricht MEMS 2004 Technical Digest, 25–29 January 2004, IEEE, Piscataway, NJ, 2004, p. 149.
[22] R.E. Boroch, J. Bagdahn, Scripta Materialia 59 (2008) 936–940.
[23] M.J. Demkowicz, A.S. Argon, D. Farkas, M. Frary, Philosophical Magazine 87 (2007) 4253–4271.
[24] C.L. Muhlstein, S.B. Brown, R.O. Ritchie, Journal of Microelectromechanical Systems 10 (2001) 593.
[25] J.E. Bradby, J.S. Williams, J. Wong-Leung, M.V. Swain, P. Munroe, Applied Physics Letters 77 (2000) 3749.
[26] A.M. Minor, E.T. Lilleodden, M. Jin, E.A. Stach, D.C. Chrzan, J.W. Morris Jr., Philosophical Magazine A 85 (2005) 323.
[27] J.P. Hirth, J. Lothe, in: Theory of Dislocations, second ed., John Wiley, New York, 1982.
[28] K. Sumino, in: T.S. Moss (Ed.), Handbook on Semiconductors, Elsevier Scientific Press, New York, 1994.
[29] O.N. Pierron, C.L. Muhlstein, Journal of Microelectromechanical Systems 16 (2007) 1441.