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Indentation Fracture Response of Al–TiN Nanolaminates

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Indentation fracture experiments on aluminium-titanium nitride nanolaminates were conducted both inside and outside of a scanning electron microscope (SEM). Remarkably, indentation fracture toughness *increases* with *increasing* strength for bilayer thicknesses less than 10 nm. In addition, slower strain rates favour formation of lateral cracking while increasing rates favour formation of radial cracks. SEM movies show that an increase in radial crack length does not occur during the unloading cycle; this is due to flow of aluminium into the cracks during unloading and is a form of self-healing which should be applicable to metal-ceramic nanolaminates in general.

Keywords: Fracture Toughness, Indentation, Thin Film

Introduction A single material rarely comprises both high strength and high toughness. Therefore, composites are traditionally used for applications that require superior values for both properties. But what happens when this combination of properties is necessary at the small length scales common in micro- and nanotechnology? In many cases an increase in strength is observed as length scales decrease, e.g. in thin films [1], micropillars [2], freestanding nanostructures [3] and nanoparticles [4] to name a few. This increase in strength generally results in a corresponding decrease in toughness that translates into the unpredictable or stochastic mechanical response common at these scales [5–7]. The increase in strength (and associated decrease in toughness) is due to a lack of mobile defects in the small volume under stress while the unpredictability is due to their random spatial distribution. Therefore, at small length scales ductile materials can behave as if they were brittle because the stress necessary to initiate plasticity (yield stress) is much greater than the stress necessary to continue the deformation (flow stress). When this situation takes place at the nanoscale, yield can occur as a displacement burst that is associated

with creation of new surface area. This event can be analogous to fracture since it produces very low strain energy release rates even in the most ductile of metals such as gold [5]. In some cases it is also possible to increase toughness at small length scales [8,9]; many times this increase in toughness depends on stress state that can include high hydrostatic pressures. Here we show that it is also possible to purposefully engineer a material with a structure that does this inherently.

The motivation behind this work is to create a composite material that has both high strength and high toughness at the nanoscale all the while exhibiting predictable yield and plastic flow behaviour. Nanolaminates are well suited for this and are a rather recent class of material; the term ‘nanolaminate’ first appeared in the mid-1990s and only gained popularity in the past decade [10]. Their structure can be either thermodynamically unstable [11] or amazingly stable [12] due to the interface structure that begins to dominate mechanical response at these small length scales. Recent work on nanolaminates composed of alternating layers of Al and TiN have exhibited co-deformation of the ceramic layers

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with the metal layers as layer thickness decreases below 5 nm [13]. In micropillar compression tests of this material the deformation is initially elastic in both the Al and TiN components. The TiN remains elastic to strains of up to 5%. This is possible because the Al component initiates plasticity via a confined layer slip (CLS) mechanism with work-hardening rates between 30% and 50% of its elastic modulus at strains between 2% and 5%. These extremely high work-hardening rates approach the theoretical maximum for thin films [14]. This type of behaviour is also seen in Al-SiC nanolaminates [15]. In addition, nanolaminates can be tolerant to radiation damage since the interfaces act as sinks for defects that normally accumulate in coarse-grain materials [16].

In order to quantify deformation and fracture at the small length scales that are important in the Al-TiN nanolaminates it is necessary to characterize the material at the same length scale, thus the applicability of nanoindentation. Indentation has been used to quantify the hardness, elastic modulus and fracture toughness of materials for many decades. Its history in the USA can be traced back to use by the US Army in the 1850s for non-destructive hardness measurements of cannon barrels using self-similar, hard indenter tips during the run up to the US Civil War [17]. However, it was not until the 1970s that the first *in situ* indentation fracture toughness experiments were conducted using an indenter combined with an inverted optical microscope positioned such that the tip-sample contact, deformation and crack formation could all be viewed from below the sample [18]. Crack growth during the full load/unload cycle could therefore be monitored allowing for insights into the fracture response of these materials [19]. The two limitations for this type of experiment are the relatively large size scale necessary for optical microscopy and the requirement of an optically transparent sample.

In the work presented here indenters both inside and outside of a scanning electron microscope (SEM) are used. SEM indentation addresses the limitation of previous *in situ* techniques (although it does impose some of its own, e.g. the requirement of a conductive sample). In this way, crack growth at sub-100 nm resolution can be recorded and made into movies during the entire load/unload cycle which enables insights into the deformation and fracture mechanisms that are active in these metal-ceramic nanolaminates including the identification of unique self-healing mechanisms and the effect of strain rate on the lateral/radial fracture system.

Experimental The Al-TiN nanolaminates were used as a model metal-ceramic system in this study. They were deposited by a DC-magnetron sputtering system with a base pressure of less than 5×10^{-7} Torr. For the Al deposition, the substrate was unbiased while 300 W was applied to the Al target in an Ar atmosphere of

4 m Torr giving a deposition rate of 0.83 nm/s. The TiN was deposited using reactive sputtering of a Ti target with 300 W of power in an Ar-N₂ atmosphere using flow rates of 25 and 15 sccm, respectively, creating a deposition pressure of 6 m Torr. This produced a deposition rate of 0.06 nm/s for TiN. The laminates were all 10 μ m thick and deposited at room temperature on {001}-silicon substrates. Four nanolaminates with different bilayer thicknesses were tested in this study; Al-TiN (90–10 nm), Al-TiN (9–1 nm), Al-TiN (5–5 nm) and Al-TiN (2–2 nm). In each case, Al was the top layer. Further details on their fabrication and stoichiometry can be found in References [13,20].

The structure of the nanolaminate is columnar; typically the column diameter is of the order of 100 nm, significantly larger than the individual layer thickness. When bi-layer thicknesses are under 10 nm, each column can be considered to be a super-lattice since within an individual column both Al and TiN exhibit {111}-type growth where the TiN grows epitaxially on the underlying Al layer with the same orientation [21].

A total of three nanoindenters were used in this work. An SEM *in situ* nanoindenter initially developed by Rabe and co-workers at Empa-Thun [22] was used to image the indentation process. Additionally, two standard indenters available at the Center for Integrated Nanotechnologies at Los Alamos National Laboratory (LANL), a Hysitron TriboIndenter[®] with a high-load option and an Agilent Nanoindenter XP[®], were also used. For indentation fracture testing a cube-corner geometry diamond tip was used in all cases in order to drive radial fracture at the relatively small loads and displacements necessary when dealing with thin films. When possible the tests were run at a constant indentation strain rate defined by Lucas et al. [23] as:

$$\dot{\epsilon}_i = \frac{\dot{h}}{h} = \frac{\dot{P}}{2P}, \quad (1)$$

where $\dot{\epsilon}_i$ is the indentation strain rate, $\dot{h}$ is the instantaneous displacement rate, h is the instantaneous displacement, $\dot{P}$ is the instantaneous loading rate and P is the instantaneous load. Using this relationship it was possible to access indentation strain rates from 10^1 to 10^{-3} s^{-1} .

In order to estimate the indentation fracture toughness of these laminates the equation proposed by Lawn et al. [24] was used:

$$K_R = \xi_{c.c.} \left(\frac{E}{H} \right)^{1/2} \left(\frac{P_{\max}}{c^{3/2}} \right), \quad (2)$$

where K_R is equal to the indentation toughness as long as the final radial crack configuration is in mechanical equilibrium with residual stress field. The geometric constant for indentation fracture using a cube corner geometry tip, $\xi_{c.c.}$, has been shown by Pharr [25] to be approximately 0.04. Both elastic modulus, E , and hardness, H , are determined using the Oliver-Pharr method [26] prior to crack

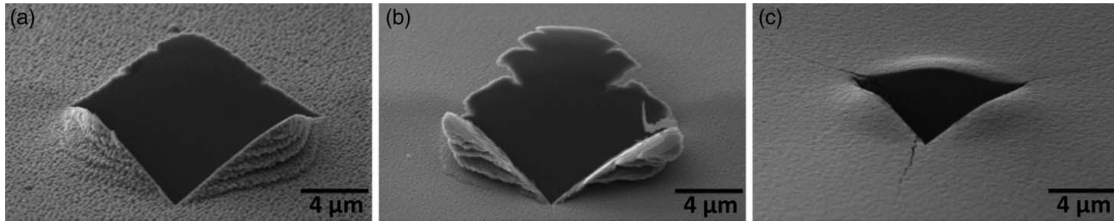


Figure 1. SEM images of indentations into the various nanolaminates, where (a) is Al–TiN (90–10 nm), (b) is Al–TiN (9–1 nm) and (c) is Al–TiN (5–5 nm). An image for Al–TiN (2–2 nm) is not shown but is similar to the (5–5 nm) laminate. All indents shown here were conducted at an indentation strain rate of 10^{-2} s^{-1} as defined in Equation (2).

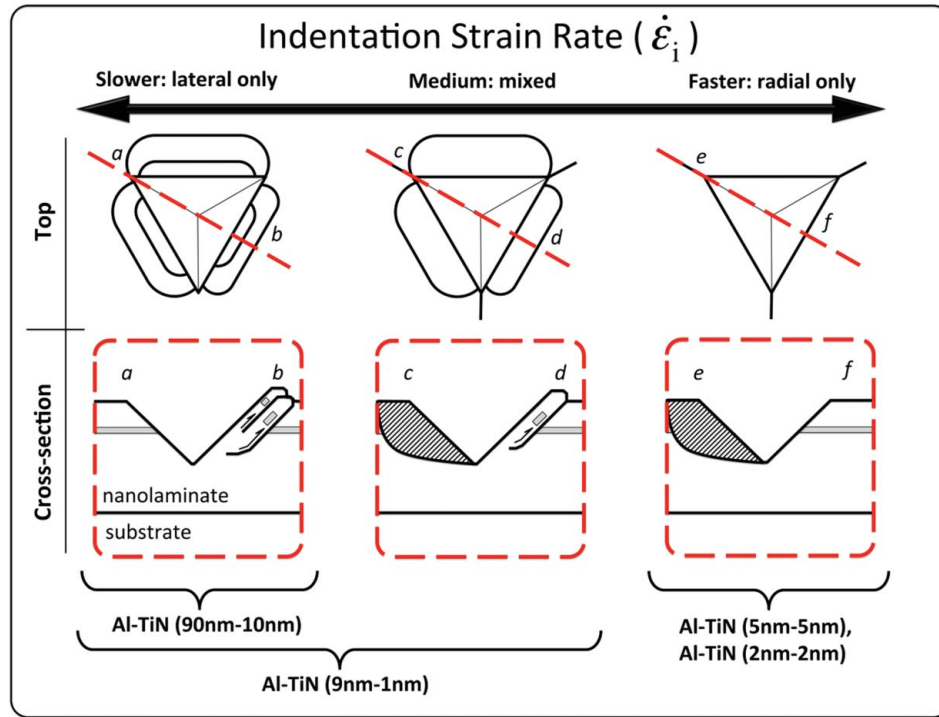


Figure 2. Schematic of the Al–TiN nanolaminate response to indentation strain rate, $\dot{\epsilon}_i$, when indented with a cube-corner geometry tip. The (90–10 nm) laminate shows only shear bands as indicated at point *b* above while there is no cracking at point *a*. Both the (2–2 nm) and (5–5 nm) laminates show only radial fracture indicated by the hatched region at point *e*. Much slower strain rates would produce shear bands. The (9–1 nm) laminate shows a transition at a strain rate of less than 10^{-3} s^{-1} from only shear bands to a mixed lateral/radial system at higher strain rates.

generation while $P_{\max}$ is the maximum load and c is the crack length as measured from the centre of the indent to the end of the surface trace of the crack determined via plan-view SEM images.

Results and Discussion The differences in the indentation response of Al–TiN of different bilayer periods during similar loadings are shown in Figure 1, where each indent reached approximately $6 \mu\text{m}$ in maximum depth and was conducted at an indentation strain rate of approximately 10^{-2} s^{-1} . Both the pile-up and cracking are vastly different for each nanolaminate at this indentation strain rate; however, the behaviour is actually consistent in that each laminate exhibits a segment of the indentation-induced lateral/radial fracture system. A schematic of the full fracture system is depicted in Figure 2.

It is currently thought that given a wide enough range of indentation strain rates, each nanolaminate would exhibit the entire lateral/radial fracture behaviour depicted in Figure 2. However, due to the limited strain rates available, each nanolaminate exhibits only a portion of the fracture system as will be described below.

Beginning with the largest bilayer thickness, the Al–TiN (90–10 nm) shows only shear band formation along the face of the indenter, an example of which can be seen in Figure 1(a) and in the supplemental movie (90 nm10 nm.mpg). These shear bands can also be thought of as lateral fracture planes. As the strain rate is decreased the spacing between shear bands in the Al–TiN (90–10 nm) laminate decreases as well. If the strain rate were orders of magnitude higher, for example, a ballistic rate of the order of 10^6 s^{-1} , radial fracture would most

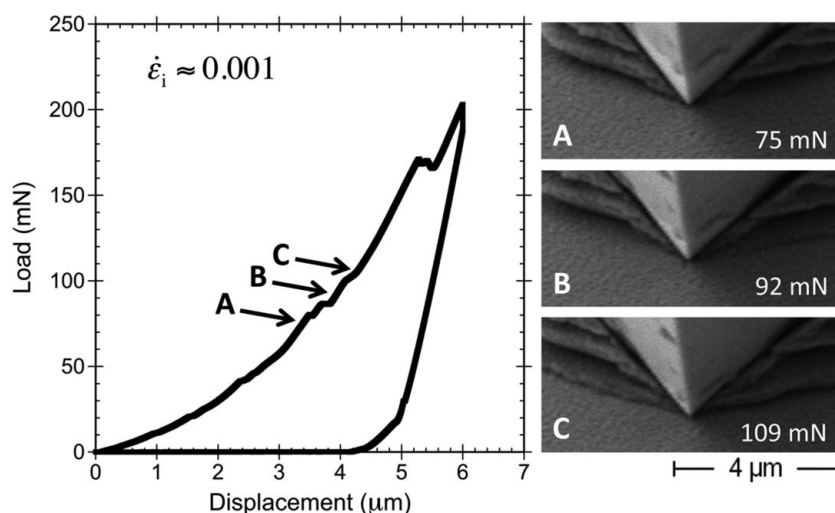


Figure 3. Load-displacement curve and corresponding SEM images of the indent from the Al-TiN (9–1 nm) nanolaminate. Slow strain rate favours formation of lateral fracture, i.e. shear bands. Image A shows no radial crack at 75 mN. By 92 mN an embryonic radial crack can be seen. This radial crack is consumed by the shear band that forms on the left-hand face of the indenter by 109 mN. Permanent radial fracture occurs at 170 mN in conjunction with the large displacement burst above. This video is available online.

likely occur. For example, fracture and spalling of pure Al occur at these rates, albeit at larger length scales [27]. Since radial fracture was not possible for this bilayer thickness, the concept of indentation fracture toughness as defined by Equation (2) was not applicable in this case.

At the other end of the lateral/radial fracture system is the behaviour of both the Al-TiN (2–2 nm) and (5–5 nm) nanolaminates depicted in Figure 2. Here, both laminates show only radial cracking for indents up to 6 μm in depth; an example can be seen in the supplemental movie entitled 5 nm5 nm.mpg. It is thought that much slower strain rates would be necessary for lateral shear band formation for these nanolaminates. For example, rates of the order of 10^{-10} , such as those active in plate tectonics, would produce lateral cracking. At the strain rates available there was no change in fracture toughness since there was no change in deformation mechanism. However, this is not the case for the Al-TiN (9–1 nm) nanolaminate.

By far the most complex and interesting deformation is exhibited by the Al-TiN (9–1 nm) nanolaminate. At a strain rate of approximately 10^{-3} both lateral and radial fracture mechanisms are active as can be seen in the movie 9 nm1 nm.mpg. Three frames have been extracted from the movie and are displayed in Figure 3 along with the relevant load-displacement curve; the first frame is at a load of 75 mN, the second at is 92 mN and the third is at 109 mN.

In the first frame at 75 mN there is no radial crack. By 92 mN two events have taken place; first a shear band begins to form on the right face of the indenter; second, an embryonic radial crack is beginning to form at the tip's apex. This crack is stable and does not extend. What happens is the shear band to the right

continues to grow and one on the left face of the indenter forms by 109 mN. The formation of the left shear band actually consumes the embryonic radial crack. A permanent radial crack does not form until 170 mN; this corresponds to a rather large displacement excursion on the load-displacement curve. For the Al-TiN (9–1 nm) nanolaminate, a strain rate lower than 10^{-3} s^{-1} exhibits only lateral shear bands while an increase in strain rate decreases the load necessary for permanent radial crack formation by about half. It is clear that shear bands are forming at the expense of radial crack formation as the strain rate decreases. This behaviour increases the laminate's apparent indentation fracture toughness as defined in Equation (2) by roughly 50% when the strain rate decreases from 1 to 10^{-3} s^{-1} as can be seen in Figure 4. As noted by Cook and Roach [28] lateral fracture planes act to decrease the constraint imposed on the plastic deformation zone by the elastic matrix through decohesion and uplift of the plastically deformed material. The slower strain rates favour lateral shear bands and therefore radial fracture is decreased. For thin films, strain rate has been reported to influence the flow stress of nanocrystalline metals [29,30], and in future work we will be investigating the effect of strain rate on the fracture of metal-ceramic nanolaminates in stress states other than indentation.

When comparing the indentation fracture toughness data taken at a strain rate of 10^{-3} s^{-1} in Figure 4 with the hardness of these films reported in a previous study [13], the indentation fracture toughness of Al-TiN nanolaminates with bi-layer thicknesses less than 10 nm actually *increases* in conjunction with indentation hardness as seen in Figure 5. An increase in hardness (and strength) as length scale decreases is rather ubiquitous as mentioned above in the Introduction. In particular, for metal-ceramic

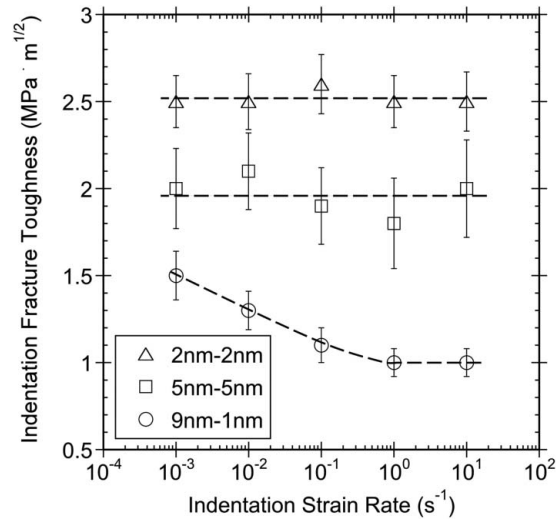


Figure 4. Indentation fracture toughness as defined by radial fracture for laminates with bilayer thicknesses less than 10 nm. Error bars represent one standard deviation. Both Al–TiN (2–2 nm) and (5–5 nm) are insensitive to strain rates in this range. The (9–1 nm) nanolaminate shows a 50% increase in toughness as strain rate decreases from 10^1 to 10^{-3} s $^{-1}$ due to an increased propensity to form lateral shear bands which suppresses radial fracture.

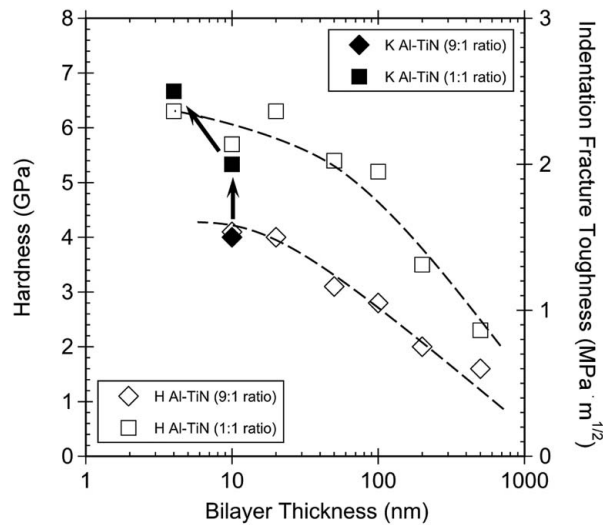


Figure 5. Measures of indentation fracture toughness (solid symbols associated with right y-axis) at a strain rate of 10^{-3} scales with hardness (open symbols associated with left y-axis) for laminates with bilayers less than 10 nm in thickness indicating that *stronger* can be *tougher* at the nanoscale.

nanolaminates this increase in hardness can be related to changes in the dominant dislocation mechanism in the metal component as bi-layer thickness decreases [31]. When the Al component has a thickness greater than approximately 30 nm, dislocations form pile-ups within the Al. During this process, the TiN is mainly undergoing elastic deformation. As the Al layer thickness decreases to below ≈ 30 nm, pile-ups are not possible and dislocations are accommodated by the CLS mechanism. The Al is confined by the TiN that is still undergoing only elastic deformation. This confinement generates the high

work-hardening rates in the Al and CLS is the dominant mechanism for Al thicknesses in the range from 30 nm down to approximately 5 nm at which point the stresses necessary for dislocation motion within the aluminium becomes so high that it is energetically favourable for the dislocations to transmit across the Al–TiN interface, enabling co-deformation of the metal and ceramic layers [32].

While the increases in hardness and strength described above are well understood, the mechanisms behind the increase in indentation fracture toughness as length scale decreases are rather obscure. This is because fracture deals with long-range interactions of unit-deformation processes while strength can be modelled by considering the individual processes. Therefore, the following discussion must include some conjecture. The question is, do the dislocation mechanisms that control strength and hardness also control indentation fracture toughness? The confinement of a ductile, work-hardening layer between ceramic layers that are undergoing only elastic deformation presents an interesting situation once cracks are introduced into the ceramic. After confinement of the aluminium has been breached, the aluminium acts to plastically flow to blunt crack tips, which in some cases involves flow into the crack. Similar behaviour has been seen in Al–SiC nanolaminates [33] where both experiments and finite element simulations of the indentation process show that the aluminium layer continues to deform during both the loading and unloading processes. Figure 6(a) shows an SEM image of an indent into the Al–TiN (5–5 nm) nanolaminate. This indent was originally loaded to 1 N that equated to a depth of 9 μ m. It was then unloaded and followed by a reload. The cracks were formed during the original loading and

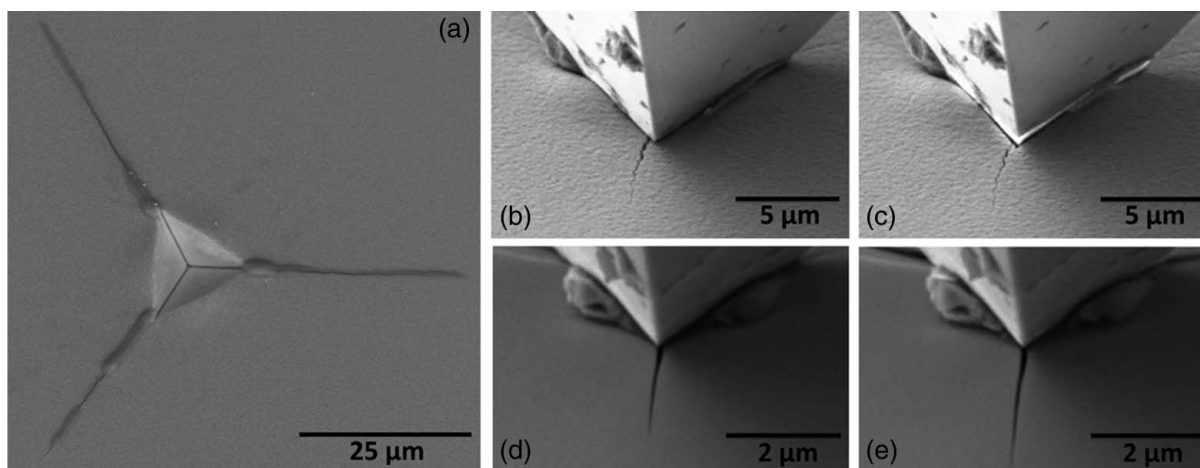


Figure 6. Self-healing mechanism active in metal-ceramic nanolaminates: (a) A large indent into the (5–5 nm) laminate (1 N maximum load) which was then unloaded followed by a reload. In this case, aluminium can be seen to have filled the cracks. Cracks do not grow upon unloading in the (5–5 nm) laminate as can be seen by comparing (b) which is taken at the maximum load (318 mN) for a smaller indent to (c) where the indenter tip is completely unloaded. For a semi-brittle material like silicon this is not the case since cracks do extend upon unloading as can be seen when comparing (d) which is an indent into Si (001) with a load of 102 mN compared to (e) which is during unloading at 20 mN.

then sometime during the unloading/reloading process; aluminium flowed into the cracks effectively self-healing the radial fractures. Aluminium flow will obviously lead to a tougher film and explains why cracks do not extend upon unloading as can be seen when comparing the SEM-indentation images taken from the supplemental movie of the Al–TiN (5–5 nm) indent at maximum load in Figure 6(b) to the same indent directly after unloading in Figure 6(c). To compare, an example of crack extension in silicon during unloading of an indent is shown in Figure 6(d) and 6(e); a movie of this indent is available in the supplemental information and is entitled Si.mpg. This type of extension is very common in traditionally brittle materials [34].

The aluminium reflow mechanism can explain toughening for both the Al–TiN (5–5 nm) and (9–1 nm) laminates. However, in the case of Al–TiN (2–2 nm) laminate co-deformation of TiN with Al may be the key factor in suppression of crack initiation.

Conclusions Indentation fracture experiments on Al–TiN nanolaminates with bilayer thicknesses of less than 10 nm show that indentation fracture toughness, as measured from the length of the radial cracks, *increases* with *increasing* strength and hardness at *decreasing* layer thicknesses. The (5–5 nm) and (2–2 nm) laminates have toughness values of approximately 2.0 and 2.5 MPa·m^{1/2}, respectively. These values are not sensitive to indentation rate in the range of 10¹–10^{–3} s^{–1}. The (9–1 nm) laminate does show a 50% increase in toughness (from 1.0 to 1.5 MPa·m^{1/2}) as indentation rates decrease from 10¹ to 10^{–3} s^{–1} due to the increased shear band formation at the expense of radial cracking at lower strain rates. In all cases, radial crack extension upon unloading was not

observed. It is thought that this is due to the flow of aluminium into the crack tip. This is a form of self-healing which should be applicable to metal-ceramic nanolaminates in general. For layer thickness greater than 10 nm, only shear bands were observed (no radial cracks) and hence indentation toughness values (from the Lawn *et al.* equation) could not be estimated.

Supplementary online material A more detailed information on experiments is available at <http://dx.doi.org/10.1080/21663831.2013.783515>.

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References

- [1] Jungk JM, Mook WM, Cordill MJ, Chambers MD, Gerberich WW, Bahr DF, Moody NR, Hoehn JW. Length-scale-based hardening model for ultra-small volumes. *J Mater Res.* 2004;19(10):2812–2821.
- [2] Maeder X, Mook WM, Niederberger C, Michler J. Quantitative stress/strain mapping during micropillar compression. *Philos Mag.* 2011;91(7–9):1097–1107.
- [3] Mook WM, Lund MS, Leighton C, Gerberich WW. Flow stresses and activation volumes for highly deformed nanoposts. *Mater Sci Eng A.* 2008;493(1–2):12–20.
- [4] Deneen Nowak J, Mook WM, Minor AM, Gerberich WW, Carter CB. Fracturing a nanoparticle. *Philos Mag.* 2007;87(1):29–37.
- [5] Mook WM, Niederberger C, Bechelany M, Philippe L, Michler J. Compression of freestanding gold nanostructures: from stochastic yield to predictable flow. *Nanotechnology.* 2010;21(5):055701-1–055701-9.

- [6] Morris J, Bei H, Pharr G, George E. Size effects and stochastic behavior of nanoindentation pop. *Phys Rev Lett*. 2011;106(16):165502-1–165502-4.
- [7] Ng KS, Ngan AHW. Stochastic nature of plasticity of aluminum micro-pillars. *Acta Mater*. 2008;56(8):1712–1720.
- [8] Gerberich WW, Mook WM, Carter CB, Ballarini R. A crack extension force correlation for hard materials. *Int J Fract*. 2008;148(2):109–114.
- [9] Beaber AR, Nowak JD, Ugurlu O, Mook WM, Girshick SL, Ballarini R, Gerberich WW. Smaller is tougher. *Philos Mag*. 2011;91(7–9):1179–1189.
- [10] Barsoum MW. The MAX phases: a new class of solids. *Prog Solid State Chem*. 2000;28:201–281.
- [11] Kim JS, LaGrange T, Reed BW, Knepper R, Weihs TP, Browning ND, Campbell GH. Direct characterization of phase transformations and morphologies in moving reaction zones in Al/Ni nanolaminates using dynamic transmission electron microscopy. *Acta Mater*. 2011;59(9):3571–3580.
- [12] Han WZ, Carpenter JS, Wang J, Beyerlein IJ, Mara NA. Atomic-level study of twin nucleation from face-centered-cubic/body-centered-cubic interfaces in nanolamellar composites. *Appl Phys Lett*. 2012 Jan;100(1):011911-1–011911-4.
- [13] Bhattacharyya D, Mara NA, Dickerson P, Hoagland RG, Misra A. A transmission electron microscopy study of the deformation behavior underneath nanoindenters in nanoscale Al–TiN multilayered composites. *Philos Mag*. 2010;90(13):1711–1724.
- [14] Nix WD. Yielding and strain hardening in metallic thin films on substrates: an edge dislocation climb model. *Math Mech Solids*. 2009;14(1–2):207–219.
- [15] Singh DRP, Chawla N, Tang G, Shen YL. Micropillar compression of Al/SiC nanolaminates. *Acta Mater*. 2010;58(20):6628–6636.
- [16] Wang J, Misra A. An overview of interface-dominated deformation mechanisms in metallic multilayers. *Curr Opin Solid State Mater Sci*. 2011;15(1):20–28.
- [17] Officers of the Ordinance Department. Hardness of metals. In: Baird HC, editor. Reports of experiments on the strength and other properties of metals for cannon. Philadelphia: US Army; 1856. p. 259.
- [18] Lawn BR, Wilshaw R. Review of indentation fracture: principles and applications. *J Mater Sci*. 1975;10:1049–1081.
- [19] Morris DJ, Cook RF. Radial fracture during indentation by acute probes: I, description by an indentation wedging model. *Int J Fract*. 2005;136(1–4):237–264.
- [20] Bhattacharyya D, Mara NA, Hoagland RG, Misra A. Nanoindentation and microstructural studies of Al/TiN multilayers with unequal volume fractions. *Scr Mater*. 2008;58(11):981–984.
- [21] Bhattacharyya D, Liu X-Y, Genc A, Fraser HL, Hoagland RG, Misra A. Heterotwin formation during growth of nanolayered Al–TiN composites. *Appl Phys Lett*. 2010;96(9):093113-1–093113-3.
- [22] Rabe R, Breguet JM, Schwaller P, Stauss S, Haug FJ, Patscheider J, Michler J. Observation of fracture and plastic deformation during indentation and scratching inside the scanning electron microscope. *Thin Solid Films*. 2004;469–470:206–213.
- [23] Lucas BN, Oliver WC, Pharr GM, Loubet J-L. Time dependent deformation during indentation testing. *Mater Res Soc Symp Proc*. 1997;436:233–238.
- [24] Lawn BR, Evans AG, Marshall DB. Elastic/plastic indentation damage in ceramics: the median/radial crack system. *J Am Ceram Soc*. 1980;63(9–10):574–581.
- [25] Pharr GM. Measurement of mechanical properties by ultra-low load indentation. *Mater Sci Eng A*. 1998;253:151–159.
- [26] Oliver WC, Pharr GM. An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. *J Mater Res*. 1992;7:1564–1583.
- [27] Pedrazas NA, Worthington DL, Dalton DA, Sherek PA, Steuck SP, Quevedo HJ, Bernstein AC, Taleff EM, Dittmire T. Effects of microstructure and composition on spall fracture in aluminum. *Mater Sci Eng A*. 2012;536:117–123.
- [28] Cook RF, Roach DH. The effect of lateral crack growth on the strength of contact flaws in brittle materials. *J Mater Res*. 1986;1(4):589–600.
- [29] Jonnalagadda K, Karanjgaokar N, Chasiotis I, Chee J, Peroulis D. Strain rate sensitivity of nanocrystalline Au films at room temperature. *Acta Mater*. 2010;58(14):4674–4684.
- [30] Karanjgaokar NJ, Oh CS, Lambros J, Chasiotis I. Inelastic deformation of nanocrystalline Au thin films as a function of temperature and strain rate. *Acta Mater*. 2012;60(13–14):5352–5361.
- [31] Misra A, Hirth JP, Hoagland RG. Length-scale-dependent deformation mechanisms in incoherent metallic multilayered composites. *Acta Mater*. 2005;53(18):4817–4824.
- [32] Bhattacharyya D, Mara NA, Dickerson P, Hoagland RG, Misra A. Compressive flow behavior of Al–TiN multilayers at nanometer scale layer thickness. *Acta Mater*. 2011;59(10):3804–3816.
- [33] Singh DRP, Chawla N, Tang G, Shen YL. Anomalous viscoplasticity during nanoindentation of Al/SiC nanolaminated composites. *Mater Sci Eng A*. 2011;528(13–14):4608–4614.
- [34] Cook RF, Pharr GM. Direct observation and analysis of indentation cracking in glasses and ceramics. *J Am Ceram Soc*. 1990;73(141):787–817.