# Positive drift post nanoindentation in titanium diboride films

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ABSTRACT

The positive drift at the end of nanoindentation P-h plot in case of hard titanium diboride ($TiB_2$) films was ascribed to swelling taking place due to strain gradient plastic deformation with the sample contacting the indenter. The indentations performed at lower loads based on the positioning however suffered resistance towards any plastic deformation as per GTN theory. Although the formation of nanocrystallites inside larger grains were observed, indentation performed at grain boundaries or area having comparatively lower atomic density caused plastic deformation.

KEWORDS: Titanium diboride, hard coating, strain-gradient, GTN

INTRODUCTION

The load-depth (P-h) plot during nanoindentation contain a whole lot of information and can unleash novel phenomenon useful in thin film studies [1]. A meticulous analysis to get a proper understanding of the surface topographical features especially related to film fracture has been previously reported in details [2-5]. However, there are some areas which requires a bit more research and shall add up to the previous existing knowledge. Titanium diboride ($TiB_2$) which is one of the well acclaimed hard coatings used for protection of components due to its high hardness and wear resistance has been taken into consideration in this case. The analysis of different nanoindentation based cracking phenomenon have been reported recently [6]. An attempt however is made in this communication to investigate a very specific aspect of linearly unloading observed at the end of the P-h plot, an area not given justified importance that it deserves.

MATERIALS & METHODS

The titanium diboride films were deposited on Si (100) substrates by magnetron sputtering using a sintered $TiB_2$ target in Ar atmosphere. Nanoindentation (MTS, USA) was performed to study the mechanical properties. The indentations were performed at two different loads and the features obtained were correlated with strain gradient. Atomic force microscope (SEIKO 400 Japan) was used for getting the microstructural features.

RESULTS & DISCUSSIONS

The ($P_{max}$, $h_{max}$) values were (0.9 mN, 50 nm) and (2.5mN, 90 nm) for the two indentations performed. A negative linear unloading has been reported due to coating delamination where the indenter moves to lower depth almost parallel to the depth axis without any associated change in load to avoid any loss of contact with the sample underneath [7]. A positive linear loading observed in both the indents on the other hand indicates swelling to have occurred where there is a centralized pileup which gets in touch with the indenter even after full load withdrawal. The indentation although starting from 0 mN may not comeback to the same state after the completion of the indentation process. The load difference ($\Delta P$) between the initial and final state can be taken as the residual load.

The unloading was higher (Δh ~ 5nm) in case of lower loads with no residual load and close to 1 nm in case of higher load indent with considerable amount of residual load. The residual load (ΔP = 0.25mN) can be related to the compressive residual stress to have developed post indentation in conjunction with previously existing film stress due CTE mismatch with the substrate. There was also a sharp change (decrease) in stiffness at 70 nm (indicating damage) which is the same where a deviation from the parabolic nature of the plot occurred. The holding time was absent in both the instances. The hardness plot corresponding to the lower indentation is given in **Fig 1d** which reached a maximum was of 20 GPa. The first contact of the film with the indenter got established at 20 nm. The $TiB_2$ film hardness previously reported was around 40 GPa in nanoindentation implying that the indentation performed was shallow and therefore the saturation was also not achieved as shown by an extrapolation.

The surface of $TiB_2$ coating is shown in **Fig 2a** where the formation of large crystallites with an average diameter of 100 nm can be seen. This accounts for the high plasticity and central swelling taking place during nanoindentation. The large grains were however made up of smaller (20-30 nm) crystallites (**Fig 2b**) giving them the high hardness. The effect of formation of nanocrystallites of hard $TiB_2$ phase was felt after a penetration to certain depth, before that the elasto-plasticity prevailed in. This is quite an intriguing phenomenon can be explained because the positioning of the indentation becomes is more significant was, we are closer to the surface. Indentation taking place at the conjunction between two crystallites or grain boundaries has more elastic nature for shallow depths giving rise to pop-ins. Indentations performed on the hard crystallite is not able to create any yield and may look like elastic with the loading and unloading curves coinciding as observed in **Fig 1c**. The closure of voids and increase in material density post nanoindentation as per *Gurson Tvergaard Needleman* (GTN) theory can also be said to be the reason behind this observation **[8]**. It may be kept in mind that the growth is not two dimensional and multiple layers of atomic depositions took place and hence the indenter on coming back to its initial state of 0 mN is at the disposal of pile ups from nearby atomic cluster causing a positive drift. The diameter of the Berkovich indenter being around 100 nm is unable to distinguish the different atomic arrangements. The swelling taking place due to plastic deformation can be seen in **Fig 2c**, where the gradient in height from the sides accounts for the strain gradient plastic deformation. of the indent tallies with the explanation provided above.

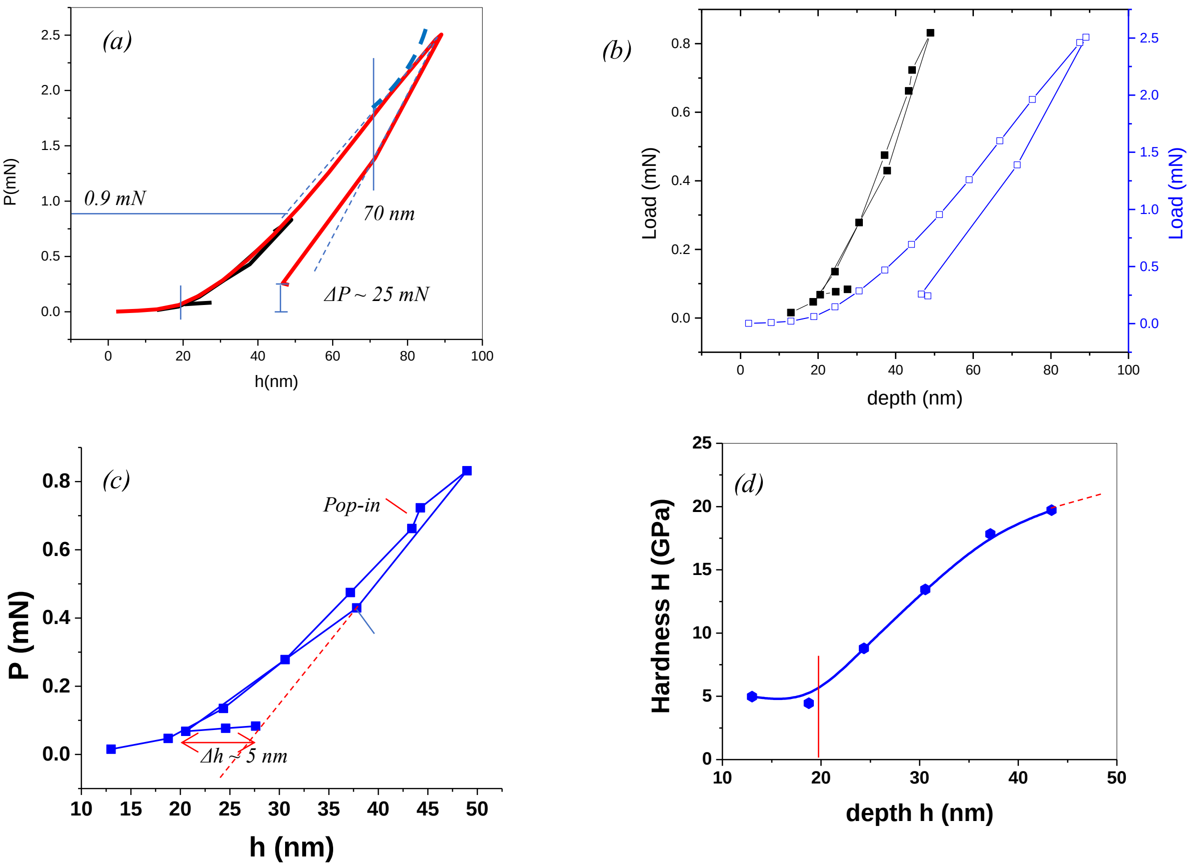


Fig 1 (a) Load-depth (P-h) plot for nanoindentation on $TiB_2$/Si coatings (a) in same load axis for two different depths (b) separate load axis (c) individual lower depth and (d) corresponding hardness.

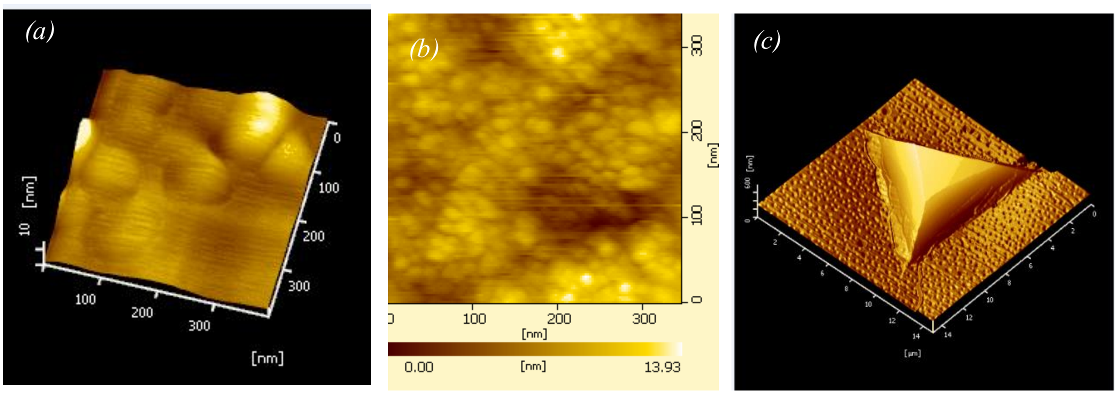


Fig 2. The surface of $TiB_2$ coating with formation of (a) large grains having (b) clustering of nanocrystallites and (c) pile-up(swelling) occurring in during nanoindentation.

CONCLUSIONS

The positive drift at the end of load depth plot during nanoindentation of $TiB_2$ film on silicon substrate was found to be governed by strain gradient plasticity. The phenomenon of close of voids and increase in material density caused by indentation was also found existing at the same time. The positioning of the indentation considering the clustering of nanocrystallites into larger grains was found significant in the overall response of the films towards nanoscale loading.

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DECLARATIONS

**Compliance with Ethical Standards**

The manuscript has not been submitted in parallel either in full or partially to any other journal.

**Conflict of interest**

There is no conflict of interest among the authors

**Research Data Policy and Data Availability Statements**

Data shall be provided on request

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