

1. Introduction

Over the years, nanocrystalline metallic multilayers (NMMs) have become a focus of extensive study [1–10] due to their superior radiation resistance and excellent mechanical performance. The NMMs are usually prepared by a layer-by-layer deposition technique. By tailoring their individual layer thickness, \( \lambda \), the NMMs exhibit length-scale-dependent hardness/strength [11–15], controlled by either the dislocation pile-up mechanism (\( \lambda \): sub-micron to micron), the confined layer slip mechanism (\( \lambda \): a few nanometres to a few tens of nanometres) or the interface crossing mechanism (\( \lambda \): 1–2 nm) [16]. Generally, peak strength of the NMMs can be achieved when the individual layer thickness reduces to a few nanometres. An example is our previous study [17] whereby a nanocrystalline Ti/Al multilayer with \( \lambda = 10 \) nm had an ultra-high specific strength, and consequently, with great potential for aerospace applications.

High creep resistance, which ensures long-term structural safety and durability, is the hallmark of aerospace materials. Due to size limitations with respect to thickness, it is nearly impossible to measure creep behaviour of the NMMs using conventional uniaxial creep testing techniques. Therefore, nanoindentation may be the only effective method to evaluate the creep properties of NMMs. Over the years, nanoindentation creep models have been developed and validated by extensive theoretical and experimental studies [18–21], and good agreements were found between the nanoindentation determined creep and conventional uniaxially determined creep. So far, the nanoindentation technique has been successfully applied to assess the creep behaviours of polymers [22–24], nanocrystalline metal alloys [25,26] and thin films [27–29]. Unfortunately, there is limited literature [30–32] concerning length-scale-dependent creep behaviour of the NMMs. Wen et al. [30] experimentally examined the creep behaviour of Ag/Co multilayers with \( \lambda = 2–30 \) nm using nanoindentation and it was found that dislocation glide-climb is a possible mechanism for the creep of Ag/Co multilayers. Zhu et al. [31] prepared Ag/Fe multilayers with \( \lambda \) ranging from 2 to 30 nm using the electron beam evaporation deposition method and the experimental results showed that the reduction of individual layer thickness resulted in a decline in the stress exponent and an increase in the creep rate of the Ag/Fe multilayers. Later, Zhu et al. [32] investigated the nanoindentation creep behaviour of Cu/Co multilayers with \( \lambda = 4–40 \) nm fabricated using an electron beam evaporation deposition method. Subsequently, a dislocation model for steady-state deformation of the NMMs with semi-coherent interfaces was presented. The individual layer thickness of the already mentioned NMMs in the studies [30–32] was less than a few tens of nanometres. To
the best of our knowledge, no previous attempts have been made to investigate length-scale-dependent creep behaviour of the NMMs with λ ranging from nano-scale to sub-micron scale.

This study aimed to experimentally investigate the length-scale-dependent nanoindentation creep behaviour of Ti/Al multilayered films with individual layer thickness ranging from nano-scale to sub-micron scale at room temperature. To do so, Ti/Al multilayers with four different individual layer thicknesses, i.e., 10 nm, 50 nm, 100 nm and 250 nm, were prepared using a direct current (DC) magnetron sputtering method. Firstly after deposition, microstructures of the Ti/Al multilayers was evaluated using transmission electron microscopy (TEM), X-ray diffraction (XRD), scanning electron microscopy (SEM), and scanning probe microscopy (SPM). After that, room temperature nanoindentation creep tests were carried out to measure the creep behaviour of the Ti/Al multilayers, such as time-dependent creep deformation under constant load, the stress exponent and the creep strain rate sensitivity. Correspondingly, the creep mechanism of the Ti/Al multilayers was discussed. Finally, contact creep compliance of the multilayers was determined and modelled by a Kelvin-Voigt model.

2. Theory

Nanoindentation creep behaviours were measured by applying a constant load on materials prior to indentation unloading. A schematic illustration of a typical indentation depth history is illustrated in Fig. 1. The indentation depth increases with creep time due to the viscoelastic behaviour of the materials. Similar to the conventional uniaxial creep, the nanoindentation creep consists of two stages, i.e., the transient creep stage, and the steady-state creep stage. In the transient stage, creep rate declines with time until a steady state is achieved whereby the creep rate appears to remain constant. The nanoindentation creep rate is defined as:

\[
\varepsilon(t) = \frac{d(t)}{d(t)}
\]  

where \( \varepsilon(t) \) is the creep rate, and \( d(t) \) is the indentation depth. In the steady-state stage, it has been reported [31] that the indentation creep response can be expressed by the following equation:

\[
\varepsilon(t) = A\sigma^n\exp(-Q/RT)
\]  

where \( A \) is a constant, \( \sigma \) is the stress, \( n \) is the stress exponent, \( Q \) is the activation energy, \( R \) is the gas constant, and \( T \) is the temperature.

To investigate the length-scale-dependent creep behaviour of the Ti/Al multilayers, three nanoindentation creep models were introduced. One of the most straightforward measures to quantify nanoindentation creep is by using a maximum creep depth, \( d_{\text{max}} \), as expressed:

\[
d_{\text{max}} = d(t_{\text{max}}) - d(t_0)
\]  

where \( d(t_{\text{max}}) \) and \( d(t_0) \) are the initial and final indentation depth during the creep time as defined in Fig. 1. The highest \( d_{\text{max}} \) indicates the lowest resistance to creep at the same load level. \( d_{\text{max}} \) is a creep indicator and has been used for parallel comparison [33].

The second method to describe nanoindentation creep behaviour is by using a phenomenological model. This model is mainly based on the work by Beake et al. [22], where they have found that the relation between the fractional increase in the indentation depth and creep time can be well fitted by a logarithmic curve as defined in:

\[
\frac{d(t)}{d(t_0)} = [m_{\text{eff}}] \ln(t/r + 1)
\]  

where \( m_{\text{eff}} \) is the creep strain rate sensitivity, which is considered as a measure of the ratio between time-dependent deformation and the deformation encountered while rapidly loading the sample to the creep load. \( r \) is the cut-off time, defined as the shortest time to produce such a creep deformation, and \( t \) is time.

An alternative method to examine the creep behaviour of the materials is by utilising contact creep compliance expressed as follows:

\[
J(t) = \frac{\varepsilon(t)}{\sigma_0}
\]  

where \( \sigma_0 \) is the stress, which is given by:

\[
\sigma_0 = \frac{P_0}{A}
\]  

where \( P_0 \) is the peak indentation load and \( A \) is the contact area. It should be noted that the contact area increases with time such that the applied stress is not a constant. However, due to the self-similar geometry of the indenter, the constant peak indentation load can be assumed to produce a constant stress [33]. Indentation of a rigid indenter to a half-space linearly viscoelastic material is a quasi-static boundary value problem with a moving boundary. Lu et al. [23] provided a general solution for this problem, given by:

\[
J(t) = \frac{\pi(1 - \nu)\tan(\alpha)}{4} \int_0^1 J(t - \xi) \left[ \frac{dP(\xi)}{d\xi} \right] d\xi
\]  

where \( \nu \) is the Poisson’s ratio of the materials, \( \alpha \) is the half-included angle of the indenter, and \( P \) is the indentation load. For a step load condition, the creep compliance \( J(t) \) has a form of,

\[
J(t) = \frac{\pi(1 - \nu)P_0\tan(\alpha)}{2d(t)^2}
\]  

From Eq. (8), we can see that the \( J(t) \) can be calculated directly from the experimentally measured \( d(t) \) for a given indenter. Based on Kelvin-Voigt model, the contact creep compliance \( J(t) \) can be fitted using the following equation:

\[
J(t) = J_0 + \sum_{i=1}^N J_i (1 - e^{-t/\tau_i})
\]  

where \( J_i \) is the compliance numbers, \( \tau_i \) is the retardation times, and \( N \) is a positive integer.
3. Material Preparation and Experimental Procedures

A DC magnetron sputtering system (Orion 5, AJA international, Scituate USA) together with 99.9% titanium and 99.9% aluminum targets were utilised to prepare Ti/Al multilayers on silicon wafers (University Wafer, USA) at room temperature. The Ti/Al multilayers were also deposited on glass substrates for XRD tests. The sputtering method was briefly discussed here. The base vacuum of the deposition chamber was maintained for at least 1 h at less than $5 \times 10^{-7}$ Torr before each deposition. The deposition pressure was set to 3 mTorr by flowing Argon gas into the deposition chamber at a rate of 10 sccm. During each deposition, the discharge power applied to each of the targets was 150 W (DC). The deposition stage was always rotated at a speed of approximately 20 rpm throughout every deposition to achieve a uniform thickness of each layer. The deposition rates of Ti and Al were approximately 4.5 nm/min and 5.3 nm/min, respectively. During the deposition of a single Ti layer, the Al target was unpowered, and its shutter was shut down. When the allotted deposition time is reached, the depositing Ti target was unpowered, and its shutter closed after which the standby Al target was powered to 150 W over 10 s with the shutter remaining shut down for ~3 min. After that, the Al layer starts to deposit with its allocated time. This cycle was repeated until the allocated layer number was achieved. Ti/Al multilayers were thus prepared with four different individual layer thicknesses; 10 nm, 50 nm, 100 nm and 250 nm. The overall thickness was estimated as ~1 μm. For all the multilayers, the final deposited layer was Ti reduce the effects of surface oxidation.

Prior to nanoindentation creep tests, surface roughness and surface grain size of the Ti/Al multilayers were identified using an SPM (Hysitron Triboindenter®, Bruker, Germany) and an SEM (Zeiss Ultra-plus, Zeiss, Germany) with an in-lens detector, respectively. The SPM scanning was performed at a scanning rate of 0.25 Hz under a setpoint of 1.5 μN. By applying a 1.5 μN load on the tested surface, it was found that the indentation depth is less than 1 nm, which is sufficiently low. The scanning area was 10 μm × 10 μm. The scanning data was further used to estimate the surface roughness of the Ti/Al multilayers. After that, the microstructures of the multilayers were detected using an XRD (Bruker D8 Advance Powder Diffractomete, Germany) and a bright-field and high-resolution TEM (JEOL 3000, Japan). XRD tests were set in θ–2θ mode using Cu-Kα radiation. Cross-sectional SEM and TEM specimens were prepared by a focused ion beam (FIB, Zeiss Auriga, Germany). Before the FIB micromachining, the selected area of the specimen was protected by depositing a platinum layer. Then, the specimen was cut at an accelerating voltage of 30 kV and a current of 250 pA. For SEM specimen preparation, the initial and final polishing was performed at 15 kV/100 pA and 15 kV/50 pA, respectively. When the TEM specimen was thinned down to ~700 nm thick, the initial and final polishing was performed at 15 kV/100 pA and 5 kV/50 pA, respectively. The final thickness of the specimen is ~300 nm. After that, the TEM specimen was lifted up by a manipulator and was attached to a copper mesh. Finally, the cross-section view of the specimen was characterised by the bright field TEM and high-resolution TEM operated at 300 kV.

The nanoindentation creep tests were conducted at room temperature (~18 °C) using a nanoindentation testing system (Hysitron Triboindenter®, Bruker, Germany with a load resolution of 1 nN and a displacement resolution of 0.04 nm. The tip function of the conical indenter was carefully calibrated in a fused quartz standard specimen and the tip function was found to be 207 nm as shown in the Appendix. The nanoindentation creep tests were carried out with a load control...
method, and thermal drifts were calibrated before each test. Loading time, creep time and unloading time were set to be 10 s, 60 s and 10 s respectively. Specifically, a peak load of 1 mN was maintained for 60 s to achieve the time-dependent nanoindentation creep. Each nanoindentation test was repeated at least ten times to obtain averaged results.

4. Results and Discussion

4.1. Microstructure

In Fig. 2, XRD spectra indicate that all of the Ti/Al multilayers are polycrystalline and contain a strong Ti (0002) and Al (111) texture. Fig. 3(a) shows the cross-sectional microstructures of the multilayer with $\lambda = 10$ nm by high-resolution TEM. Here it is observed that the interface between subsequent Ti and Al layers is relatively abrupt, which is different to the Ti/Al multilayer interfaces prepared by Ramos et al. [34]. It was also reported by Banerjee et al. [35] that phase transitions occur in both the Ti and Al layers when the individual layer thickness is reduced to a few nanometres. However, the atomic stacking sequence observed with TEM and displayed in Fig. 3(a) confirms that the Ti layers have a hexagonal close-packed structure while the Al layers exhibit a face-centred cubic structure. This orientation relationship between the Ti layer and Al layer is consistent with that measured by the XRD and is preserved for all individual layer thicknesses (see Fig. 2). To view the microstructure of the Ti/Al multilayers on a larger scale, FIB has been used to obtain the cross-section of the multilayer with $\lambda = 10$ nm. It is found that the Ti/Al layers are alternately deposited onto the silicon wafer with relatively abrupt interfaces aligned perpendicularly to the deposition direction as shown in Fig. 3(b). The overall thickness of the Ti/Al multilayers is confirmed to be ~1 $\mu$m, which is consistent with our prediction and applied Ti and Al deposition rates.

The surface morphology of the Ti/Al multilayers is examined by SPM with a setpoint of 1.5 $\mu$N, as shown in Fig. 4(a)–(d). The surface roughness $S_q$ (root mean square height) is then obtained and are listed in Table 1. It is seen that while the surface roughness of the Ti/Al multilayers increases with increasing individual layer thickness, the measured $S_q$ values lay in the range of 1.51–5.57 nm which is considered to be a relatively smooth, high-quality surface finish. Fig. 5(a)–(d) shows grain distribution on the surfaces of the Ti/Al multilayers as revealed by SEM. It is seen that all of the multilayers have relatively uniform distributions of surface grains. By analysing the SEM images, the surface grain sizes of all the Ti/Al multilayers are

<table>
<thead>
<tr>
<th>$\lambda$ (nm)</th>
<th>10</th>
<th>50</th>
<th>100</th>
<th>250</th>
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</thead>
<tbody>
<tr>
<td>$S_q$ (nm)</td>
<td>1.51</td>
<td>4.01</td>
<td>4.71</td>
<td>5.57</td>
</tr>
<tr>
<td>Surface grain size (nm)</td>
<td>211 ± 56</td>
<td>238 ± 81</td>
<td>245 ± 76</td>
<td>260 ± 81</td>
</tr>
</tbody>
</table>
It is found that all of the multilayers display similar surface grain sizes which are attributed to the use of consistent deposition conditions for all samples. The grain sizes are in the range of 211–260 nm and can be categorised as fine-grain metallic materials.

Due to the relatively smooth surface and uniform grain size, the following nanoindentation creep tests can be conducted at a relatively low load to reduce the substrate effect with expecting repeatable creep responses for each multilayer.

### 4.2. Length-Scale-Dependent Creep Behaviour

Room-temperature nanoindentation creep tests are carried out at a peak load of 1 mN with a creep time of 60 s. Typical indentation load and depth relations are obtained as illustrated in Fig. 6. It is observed that the maximum indentation depth increases with an increase in the individual layer thickness at the same load level, owing to the length-scale-dependent strength of the Ti/Al multilayers reported in our previous work [17]. It is worthwhile noting that the silicon substrate may affect the creep results of the Ti/Al multilayers because the highest indentation depth is roughly a quarter of the film thickness. To measure “film-only” properties by nanoindentation, one commonly employed rule of thumb is to limit the indentation depth to lower than 10% of the film thickness [36] when the substrate deformation is negligible. However, this method is usually unfeasible for very thin films. Increasing the film thickness is a solution to apply the “10% principle” to minimise the substrate effect. However, the deposition time significantly increases especially for the multilayered film with an individual layer thickness of 10 nm. Another method proposed by Saha et al. [37] to obtain the true film properties is to deposit the film onto a substrate with a similar modulus as that of the film to meet the deformation compatibility of film/substrate systems with various modulus ratios. In particular, when the modulus ratio of film/substrate is in the range of 0.7–1, the substrate factor is within 5% when the indentation depth is less than a quarter of the film thickness. In this study, the modulus ratio of film and substrate is ~0.72. Therefore, the substrate effect is low and can be neglected. It is found that the increase in indentation depth during the period of constant indentation load increases as the individual layer thickness also increases. This implies length-scale-dependent creep behaviour of the Ti/Al multilayers. As a function of time, the creep...
deformations of all multilayers are plotted in Fig. 7(a), and as seen, all increase over time. The multilayer with \( \lambda = 10 \text{ nm} \) shows the lowest creep deformation while the multilayer with \( \lambda = 250 \text{ nm} \) exhibits the highest. The creep rates are then determined using Eq. (1) and are shown in Fig. 7(b). It is noted that the creep deformation consists of two stages, i.e. a transient creep stage, followed by a steady-state creep stage. In the transient creep stage, the creep strain rate is relatively high in the order of \( 10^{-2} \text{–} 10^{-1} \). The creep strain rate decreases rapidly with increasing the creep time for all the multilayers. With extending the creep time, the strain rate gradually approaches a constant in the order of \( 10^{-4} \text{–} 10^{-3} \), which relates to the "steady-state creep stage". As indicated in the inserted figure, the creep rate at the steady-state creep stage changes from \( 3.66 \times 10^{-4} \) to \( 1.85 \times 10^{-3} \) as the individual layer thickness increases from \( 10 \text{ nm} \) to \( 250 \text{ nm} \). This tendency is similar to that in Ag/Fe multilayers [31]. The maximum creep deformation, \( d_{\text{max}} \) as a function of individual layer thickness has been examined and illustrated in Fig. 8. It is evident that the maximum creep depth increases with increased individual layer thickness. In other words, the multilayer with an individual layer thickness of \( 10 \text{ nm} \) shows the highest creep resistance among all the multilayers. In particular, the maximum creep deformation and individual layer thickness show a linear relationship when the individual layer thickness is no less than \( 50 \text{ nm} \). For the \( 10 \text{ nm} \) multilayer, the maximum creep deformation is lower than the linear trend which indicates a strengthened creep resistance. For the multilayers with \( \lambda \) ranging from \( 50 \text{ nm} \) to \( 250 \text{ nm} \), dislocation movement is suppressed by the interfaces between each layer, resulting in a dislocation pile-up at the interface. The relation between strength and \( \lambda \) can be described by the Hall-Petch scaling law. By incorporating the Hall-Petch scaling law into the thermally activate flow model, the following length-scale creep strain rate can be obtained [39,40],

\[
\varepsilon(t) = B \exp \left[ \frac{-Q}{RT} + C(\sigma - \sigma_0 - k\varepsilon^{-1/2}) \right] 
\]

where \( B, C \) and \( k \) are the experimental constants. \( \sigma_0 \) is the flow stress. \( \sigma \) is the single crystal internal stress. In Eq. (10), it is seen that the creep strain rate increases with an increase in the \( \lambda \), which is consistent with our results in Fig. 7(b). Additionally, for the Ti/Al multilayer with a higher creep strain rate, the increment of the creep depth over a specific time, (e.g. \( d_{\text{max}} \)) is larger. In other words, the creep strain rate is positively related to the \( d_{\text{max}} \). Therefore, the \( d_{\text{max}} \) increase with an increase in the \( \lambda \). It is difficult to theoretically determine the relation between \( d_{\text{max}} \) and \( \lambda \). However, a linear relation is found when \( \lambda \) is in the range of \( 50\text{–}250 \text{ nm} \) as shown in Fig. 8. With the reduction of \( \lambda \) to \( 10 \text{ nm} \), the confined layer slip mechanism is dominated, and the relation between strength and \( \lambda \) deviates from the Hall-Petch relation. As a result, the tendency of the relation between creep strain rate and \( \lambda \) deviates from the relation described in Eq. (10) when the individual layer thickness drops to \( 10 \text{ nm} \). This is also confirmed by the result in Fig. 8. Furthermore, we found that the deviation of maximum creep deformation in \( 250 \text{ nm} \) shows the highest deviation, which may be due to the comparatively high surface roughness of this sample as shown in Fig. 4(d).

To further explore the creep mechanism of the Ti/Al multilayers under nanoindentation, stress exponents for all the multilayers are evaluated. For an indenter with a self-similar geometry, Eq. (2) can be rewritten as [31],

\[
\ln(t) = -n \ln[H(t)] + B 
\]

where \( H(t) \) is the instantaneous hardness and \( B \) is a constant. Thus, the stress exponent can be extracted from Eq. (11) by curve fitting. Typical fitting of the \( \ln[H(t)] \) and \( \ln(t) \) together with the extracted \( n \) value for all the Ti/Al multilayers are shown in Fig. 9(a)–(d). It is found that the stress exponent increases with decreasing individual layer thickness and that the values of the stress exponent range from 15.74 to 61.46.

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**Fig. 7.** (a) Typical creep depth histories in the multilayers with different individual layer thickness and (b) creep strain rate versus creep time.

**Fig. 8.** Maximum creep depth versus individual layer thickness.
Generally, the values of the stress exponent are related to the creep mechanism. For instance, when $n = \sim 1$ the creep process is controlled by the diffusional creep mechanism [41]. When $n$ approaches 2, the creep process is dominated by the grain boundary sliding mechanism [42]. When $n$ is greater than 3, the creep mechanism is associated with dislocation movement such as dislocation glide-climb [43,44]. In this study, the lowest $n$ is 15.74, indicating that the nanoindentation creep in all of the Ti/Al multilayers is dominated by the dislocation glide-climb mechanism. When the dislocation motions are held up by the interfaces between Ti and Al layers, the dislocation is able to climb to its parallel slip plane by diffusion. For the multilayer with $\lambda = 10$ nm, the high occurrence of interfaces provides more diffusion paths for the diffusion required for dislocation climb. As such, the interfaces are acting as sources for emitting and absorbing the dislocations. Hence, the multilayer with $\lambda = 10$ nm has the highest creep resistance. In contrast, the multilayer with $\lambda = 250$ nm has the fewest interfaces and diffusion paths, and results in the lowest creep resistance.

Next, we applied a phenomenological model to examine creep strain sensitivity of the Ti/Al multilayers. The creep curves were fitted by a logarithmic equation defined in Eq. (4), as shown in Fig. 10(a). It is seen that the creep curve is well fitted by a logarithmic equation with a coefficient of determination, $R^2$, of 0.9996. The parameters creep strain rate sensitivity and cut-off time, are thereafter extracted from the fitted curves as illustrated in Fig. 10(b) and (c), respectively. Creep strain rate sensitivity is an “extent” term of the creep, indicating the creep magnitude. It is found that the strain rate sensitivity decreases with decreasing individual layer thickness, indicating a length-scale-dependent creep behaviour of the Ti/Al multilayers. At the same time, the linear relation holds well when the layer thickness is no less than 50 nm. For the multilayer with $\lambda = 10$ nm, it is clear that it deviates from linearity. The cut-off time is derived based on the phenomenological creep model proposed by the work [22]. It is the “rate” term of the creep, indicating the shortest time to produce such a creep deformation. Fig. 10(c) shows the cut-off time as a function of individual layer thickness. It is found that the cut-off time increases with an increase in the individual layer thickness. In particular, it is found that the cut-off time is the lowest among all of the multilayers. Above all, the multilayer with the lowest individual layer thickness has the highest creep resistance.

4.3. Modelling of Creep Behaviour in Ti/Al Multilayers

The creep behaviours of the Ti/Al multilayers with different individual layer thickness have been modelled using a Kelvin-Voigt model. First, the contact creep compliances for all of the multilayers are
evaluated using Eq. (8), and are illustrated in Fig. 11(a). Creep compliances for all the multilayers increase with time, and the multilayer with $\lambda = 10 \text{ nm}$ shows the highest increase rate. In addition, it is seen that the Ti/Al multilayer with $\lambda = 250 \text{ nm}$ has the highest creep compliance whereas the multilayer with $\lambda = 10 \text{ nm}$ exhibits the lowest. The variation of the creep compliances for all of the multilayers during 60 s are obtained as showed in Fig. 11(b). The variation of creep compliance increases with an increase of the individual layer thickness, in line with the results of time-dependent creep deformation. Then, the creep compliance for all of the multilayers is modelled by Kelvin-Voigt elements in series as illustrated in Fig. 12(a). Typical fitting of the contact creep compliance is shown in Fig. 12(b). In this study, we found that the creep compliance can be well fitted using Eq. (9) with $N = 2$. The fitting into Eq. (9) allows for the determination of creep numbers and retardation times. For example, the creep function for the Ti/Al multilayer with $\lambda = 250 \text{ nm}$ using a conical indenter is expressed as:

$$J(t) = 0.1011 + 0.02744(1 - e^{-t/33.22}) + 0.014(1 - e^{-t/2.886})$$

(12)

Typical fitting results for the multilayers with different individual
layer thickness are listed in Table 2. It is seen that the contact creep compliance can be well fitted by a Kelvin-Voigt element in a series model with a coefficient of determination, $R^2$, up to 0.9997. The creep number $J_i$ (where $i = 0, 1, 2$) and retardation times $\tau_j$ ($j = 1, 2$) increases with increasing individual layer thickness. This implies that the multilayers with the lowest individual layer thicknesses have the highest creep compliance. This conclusion is consistent with the previous creep results.

5. Conclusions

In the present work, the length-scale-dependent creep behaviour of Ti/Al multilayers has been investigated by nanoindentation, together with XRD and TEM techniques. The following conclusions can be drawn:

(a) The fine grain size polycrystalline Ti/Al multilayers with strong Ti (0002) and Al (111) textures were prepared by magnetron sputtering method. The interfaces between Ti and Al layers were abrupt, and the surface roughness was low.

(b) The maximum creep deformation, and creep strain rate sensitivity, increase with increased individual layer thickness, implying length-scale-dependent creep behaviour.

(c) The stress exponent is found to range from 15.74 to 61.46, indicating that the enhancement of creep resistance was controlled by a dislocation creep mechanism.

(d) Contact compliances for all the multilayers were well modelled by a two-element Kelvin-Voigt model.

Acknowledgements

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Appendix A

The tip function of the used indenter was determined by carefully calibrating in a standard fused quartz using the following equation.

$$ A_e = C_0 h_c^2 + C_1 h_c + C_2 h_c^{0.25} + C_3 h_c^{0.25} $$

where $C_0$, $C_1$, $C_2$ and $C_3$ are the constants by curve fitting, $A_e$ is the contact area, $h_c$ is the contact indentation depth. Fig. A1 shows the experimental data and the fitting curves. It is found that the tip function can be fitted well by a linear equation when the indentation depth is shallow,

$$ A_e = 2\pi R h_c $$

where $R$ is the tip radius. After fitting, $R$ has a value of 207 nm.

<table>
<thead>
<tr>
<th>Layer thickness (nm)</th>
<th>$J_0$ (1/GPa)</th>
<th>$J_1$ (1/GPa)</th>
<th>$\tau_1$ (s)</th>
<th>$J_2$ (1/GPa)</th>
<th>$\tau_2$ (s)</th>
<th>$R^2$</th>
</tr>
</thead>
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<tr>
<td>10</td>
<td>0.007579</td>
<td>0.0005397</td>
<td>7.022</td>
<td>0.0003498</td>
<td>0.5842</td>
<td>0.9247</td>
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<tr>
<td>50</td>
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<td>0.004728</td>
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<td>0.002183</td>
<td>1.754</td>
<td>0.9997</td>
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<tr>
<td>100</td>
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<td>0.009587</td>
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<td>0.004232</td>
<td>2.575</td>
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</tr>
<tr>
<td>250</td>
<td>0.1011</td>
<td>0.02744</td>
<td>33.21</td>
<td>0.014</td>
<td>2.868</td>
<td>0.9995</td>
</tr>
</tbody>
</table>

Fig. 12. (a) Schematic of Kelvin-Voigt model and (b) curve fitting of creep compliance.
Fig. A1. Curve fitting of the tip function of the indenter.

References


