Nanomechanical and Tribological Properties of Hexagonal-Boron Nitride-Enhanced Sintered Titanium Alloy Matrix Composites

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Abstract. This work explored the influence of varied content (1, 3 and 5 wt.%) of hexagonal boron nitride (h-BN) on the nanomechanical and tribological properties of Ti6Al4V matrix composites (TMCs) developed by spark plasma sintering (SPS). Scanning electron microscopy/energy-dispersive X-ray spectroscopy, optical microscopy and X-ray diffraction were employed to characterize the microstructural and phase constituents of the sintered TMCs. Also, nanoindentation and tribology experiment using an automated nanoindenter at maximum load of 200 mN and pin on disk tribometer under 5, 10 and 15 N applied loads were performed, respectively. The results obtained showed that SPS enabled accomplishment of well-refined grains, formation of highly densified product and development of solid matrix-reinforcement interfacial bond. It was also found that the TMCs exhibited continuing enhancement in nanoindentation hardness (50.66 ± 2.25-70.78 ± 3.34 GPa) and modulus of elasticity (238.69 ± 12.25-356.76 ± 21.34 GPa) values and improved tribological properties with increasing h-BN reinforcement content.

1 Introduction

The need for the development of advanced materials with superior performance properties in engineering applications remains an ever-growing quest [1]. The dual phase (α+β) titanium alloy, Ti6Al4V is the most sought-after titanium alloy for most-used energy-saving applications by manufacturers in aerospace and automobile industries due to its excellent blend of mechanical, physical and chemical properties, and a long history of application [2-3]. Even so, low hardness and poor tribological properties have restricted the extensive industrial applications of the titanium alloy [4-5]. Researchers and developers in the field have made substantial efforts and publicized that the development of ceramic particulate reinforced Ti6Al4V matrix composites (TMCs) is an effective method of achieving enhanced properties of the titanium alloy [6-7]. Among the existing ceramic particulates, hexagonal...
boron nitride (h-BN) is considered a promising reinforcement due to its unique blend of excellent lubrication properties, low density, thermal stability, chemical inertness, thermal shock resistance and non-toxicity. h-BN is a versatile engineering ceramic gaining steady prominence in aerospace, metallurgical and chemical engineering applications [8-9].

The microstructure and properties of materials depend on their processing history and technological regime [10]. With spark plasma sintering (SPS) technique, the development of nanocomposite materials with enhanced microstructural orientations and excellent physical and mechanical properties with fewer defects is achievable through rapid sintering and densification process [11]. Material’s testing of mechanical properties is very important in determining the behaviour of TMCs during their service life. For a long time, various destructive testing techniques at microscale level including hardness, tensile, elastic modulus, fracture toughness, impact, and many others, have been used to assess the mechanical properties of titanium alloys and TMCs [12]. Recently, advancement in materials research has driven the development of nanoindentation technique of mechanical testing of materials at nanoscales by using extremely low loads and depths [13]. The nanoindentation technique of mechanical properties testing has been successfully applied to diverse materials such as polymers, ceramics, alloys, compounds and composites, with many advantages. More especially, nanoindentation technique does not alter the microstructural integrity of the materials being tested and eliminate the need for bulk materials and standard geometries as often required in conventional mechanical testing. Moreover, it makes it possible to generate load-displacement data which is subsequently analyzed to obtain various mechanical properties, especially hardness and modulus of elasticity [14-15].

Therefore, in this work, the authors explored the influence of varied contents (1, 3 and 5 wt. %) of h-BN on the nanomechanical and tribological properties of TMCs developed by SPS.

2 Methodology

The matrix, Ti6Al4V ASTM Grade 5 powder of average particle size of 60 µm and reinforcement of nano h-BN powder consisted of average particle size of 100 nm, both of >99% purity, were provided by TLS-Technik GmbH & Co (Germany) and Hongwu International Group Ltd. (China), respectively. The transmission electron microscopy (TEM) and X-ray diffraction (XRD) images of the supplied powders are displayed in Fig. 1.

Each quantity of powder required to produce the experimental samples of Ti6Al4V alloy and TMCs was calculated using the principle of the rule of mixtures by applying equations (1) and (2), respectively. The details of the result are presented in Table 1 [16-18].

\[
\frac{1}{Dt} = \frac{Wm}{Dm} + \frac{Wr}{Dr}
\]

(1)

\[
W = Dt \times \pi r^2 h
\]

(2)

where \(Dt\), \(Dm\) and \(Dr\) are the theoretical densities of the TMC, Ti6Al4V alloy matrix (4.43 g/cm\(^3\)) and h-BN reinforcement (2.3 g/cm\(^3\)), respectively; \(Wm\) and \(Wr\) are the weight fractions of the Ti6Al4V alloy matrix and h-BN reinforcement, respectively; \(W\) is the quantity (in gram) of the TMC powder to be loaded into the prepared die assembly and \(\pi r^2 h\) is the volume of the experimental sample of dimensions 30mm diameter by 10mm height.
Table 1. Quantity of powders measured using the principle of the rule of mixture.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Powder composition</th>
<th>Mass of Ti6Al4V (g)</th>
<th>Mass of h-BN (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti6Al4V</td>
<td>100 wt. % Ti6Al4V</td>
<td>31.32</td>
<td>0</td>
</tr>
<tr>
<td>Ti6Al4V-1h-BN</td>
<td>99% wt. Ti6Al4V-1 wt. h-BN</td>
<td>30.73</td>
<td>0.31</td>
</tr>
<tr>
<td>Ti6Al4V-3h-BN</td>
<td>97% wt. Ti6Al4V-3 wt. h-BN</td>
<td>29.56</td>
<td>0.91</td>
</tr>
<tr>
<td>Ti6Al4V-5h-BN</td>
<td>95% wt. Ti6Al4V-5 wt. h-BN</td>
<td>28.41</td>
<td>1.50</td>
</tr>
</tbody>
</table>

The calculated powders were carefully measured in a plastic container with an electronic weighing balance of high precision accuracy of 0.001 g and subsequently blended in a tubular mixer set at the operating speed of 100 rev/min for 10 hours. By adopting the standard SPS working principles, an automated SPS machine of model HHDP-25, FCT Systeme GmbH (Germany) was used to consolidate the blended powders to produce the experimental samples in vacuum by applying an optimized sintering parameters: 1000 °C temperature, 30 MPa pressure, 100 °C/min heating rate and 10 min holding time, obtained from a previous experiment [16]. In the end, four samples were developed, and sand blasted. The density measurement of the sintered samples was done with the aid of an automatic electronic densimeter (JA5003J) of 0.001 g/cm³ resolution in accordance to ASTM B962 using Archimedes’ method. The measured density was obtained from an average of six repeated measurements while the theoretical density was calculated by applying equation (1) as earlier mentioned. After that, the relative density of the samples was calculated by applying equation (3) [16-18].

\[
\text{Relative density} = \left(\frac{\text{Measured density}}{\text{Theoretical density}}\right) \times 100\% \quad (3)
\]

Afterward, the samples were sectioned into pieces of dimensions 10 mm x 10 mm x 5 mm by wire-cutting and hot mounted by using epoxy resin. Grinding was done by using silicon carbide abrasive papers of European (P-Grade) grit sizes ranging from P120 to P2400. Polishing was achieved by using DiaMaxx (two-in-one monocrystalline diamond suspension manufactured by Akasel, Denmark) of 6, 3 and 1 microns for about 10-15 seconds each until the sample was scratch-free and mirror-like shiny. Freshly prepared Kroll’s reagent (2 ml hydrofluoric acid, 5 ml nitric acid and 100 ml water) was used to etch the samples.

The microstructure and phase characterization of sintered samples were conducted by scanning electron microscope equipped with an energy dispersive spectroscopy (SEM/EDS, TESCAN VEGA3, Czech Republic) operated by Oxford software at 20 kV acceleration voltage and an optical microscope (Olympus BX 51 TRF, Japan), respectively. For phase characterization, the X-ray diffraction (XRD) patterns of the samples were recorded by using X-ray diffractometer (XRD Bruker D8 Advance) equipped with CuKα radiation (1.54060 Å wavelength), operated at 40 kV and 40 mA and the diffraction angle (2θ) were collected between 5° and 90° performed in step size of 0.017° and scan step time of 30s. Afterward, the phase identification was performed by employing PANalytical X’Pert Highscore Plus software and Reitveld refinement approach.

The nanomechanical study of the TMCs was done by means of nanoindentation experiment according to ISO 14577 with the aid of an Anton Paar Nanoindenter (NHT) set up with a Berkovich indenter [12]. To ensure better accuracy of the test results, the nanoindenter was first calibrated using a reference of fused silica sample [13]. Afterward, the
experiment was carried out at 200 mN constant applied load and held for 20 s to enable the achievement of the time dependent deformation properties of the materials. An average of six indents on the sample’s surface selected were taken for indentation measurements of each sample. The nanoindentation test was governed by Oliver-Pharr technique, in which hardness, H (in GPa) and modulus of elasticity, E (in GPa) of the samples were determined by employing equations (4) and (5), respectively [14,19]

\[ H = \frac{P_{\text{max}}}{A_c} \]  

where Ac is the projected contact area of the nanoindenter’s tip with the sample at the maximum load \( P_{\text{max}} \)

\[ \frac{1}{E_r} = \frac{1-V_s^2}{E_s} + \frac{1-V_i^2}{E_i} \]  

where \( E_i \) and \( E_s \) are the modulus of elasticity of the nanoindenter’s tip and sample, respectively; \( E_r \) is the reduced modulus of elasticity which has taken into consideration the individual contributions of \( E_i \) and \( E_s \); whereas \( v_i \) and \( v_s \) are the Poisson’s ratios of the nanoindenter’s tip and the sample, respectively.

The tribological study of the TMCs was done at room temperature by using Anton Paar Tritec SA (Switzerland) TRB\(^3\) pin-on-disk tribometer with a rotary wear module. A counter body of 10mm diameter steel ball was employed as a roller against the samples. Normal loads of 5N, 10N and 15N were applied at 150 revolution per minute. The variations in the coefficient of friction (COF), the average COF values and the specific wear rates all through the test were assessed to determine the tribological properties of the TMCs.

3 Results and Discussion

3.1 Characterization of powder materials

The TEM images showing the spherical particles of the supplied Ti6Al4V alloy and platy flakes of \( h \)-BN are presented in Fig. 1(ai-aii) while Fig. 2(bi-bii) shows the XRD images of the two powders identified with only \( \alpha \) phase peaks at diffraction angles, \( 2\theta = 35.09^\circ, 38.42^\circ, 40.17^\circ, 53.0^\circ, 62.95^\circ, 70.66^\circ, 76.22^\circ \) and \( 77.37^\circ \) equivalent to miller indices \((100), (002), (101), (102), (110), (112)\) and \( (201) \), respectively, and high intensity peaks of \( \sim 100-200 \)nm crystalline \( h \)-BN phase at diffraction angles, \( 2\theta = 26.63^\circ, 41.51^\circ, 43.75^\circ, 50.0^\circ, 55.1^\circ, 75.73^\circ \) and \( 81.93^\circ \), matching miller indices of \((002), (100), (101), (102), (004), (110)\), and \((105)\), respectively [8].
3.2 Characterization of sintered composites

The secondary electron image (SEI) in Fig. 2(a) shows the dual (α and β) phases of a typical microstructure of spark plasma sintered Ti6Al4V alloy with different orientations of the α lamellar colonies while in the SEIs of the sintered TMCs are shown in Fig. 2(b-d). The α/β phases in Fig. 2(a) are apparently stabilized by the aluminium and vanadium contents, respectively, and backed by the EDS spectra portraying the elemental peaks of the probable amounts. The degree of distribution of h-BN nanoparticles and the influence of its contents on the microstructure of the α/β alloy matrix can be observed as substantiated in Fig. 2(b-d). Apparent microstructural transformations are noticeable in the TMCs due to the presence of h-BN reinforcement, which can be observed to increase from 1 wt. % in Fig. 2(b) to 3 wt. % in Fig. 2(c) as revealed by more concentration of h-BN particles within the α/β alloy matrix. However, clustering of the h-BN particles can be noticed at the grain boundary regions when more h-BN content (up to 5 wt. %) was added as exemplified in Fig. 2(d). Furthermore, the SEI of Ti6Al4V alloy in Fig. 2(a) reveals basically minimal porosities. This suggests that SPS enabled accomplishment of well-refined grains, formation of highly densified product
and development of solid matrix-reinforcement interfacial bond [14]. Meanwhile, with the addition of $h$-BN reinforcement, tiny pores can be observed across the TMCs with 1 and 3 wt. % reinforcements as shown in Fig. 2(b-c) while the inclusion of porosities and formation of $h$-BN clusters can be noticed in TMC with 5 wt. % reinforcement as shown in Fig. 2(d).

The plots in Fig. 3(a-b) depict the XRD patterns of the Ti6Al4V alloy matrix and $h$-BN reinforcement particles before mixing and sintering while Fig. 3(c-f) depict the XRD patterns of sintered Ti6Al4V alloy and TMCs with increasing content of $h$-BN, respectively. The XRD pattern in Figure 3(c) affirmed the presence of both $\alpha$ and $\beta$ Ti phases. The diffraction peaks of the $\alpha$ phase can be identified at about the same positions in both the sintered Ti6Al4V alloy and TMCs as depicted in Figure 3(c-f). Similarly, the XRD patterns of all the TMCs can be seen to display similar profile and no remarkable alterations in the positions of the diffraction peaks of the secondary phase particles. It is equally clearly revealed that the relatively high peak intensities of Ti6Al4V alloy and $h$-BN particles prior sintering as shown in Fig. 3(a-b) substantially diminished after sintering due to broad dispersion of the $h$-BN particulate reinforcement on the Ti6Al4V alloy matrix accompanied by microstructural and phase modifications with the formation of secondary phase particles as shown in and Fig. 3(d-f). These secondary phase particles are generally comprised of BN occurring at diffraction angle, $2\theta = 26.77^\circ$ and $41.6^\circ$, while TiB occurred at $2\theta = 42.2^\circ$, and AlN at $2\theta = 36.0^\circ$, respectively [6,20].
Fig. 2. SEIs of sintered samples: (a) Ti6Al4V alloy (b) Ti6Al4V-1h-BN (c) Ti6Al4V-3h-BN and (d) Ti6Al4V-5h-BN
Fig. 3. XRD image showing the patterns of (a) Ti6Al4V powder (b) h-BN powder (c) sintered Ti6Al4V alloy (d) Ti6Al4V-1h-BN (e) Ti6Al4V-3h-BN and (f) Ti6Al4V-5h-BN

3.3 Densification properties of sintered composites

The relative density of the sintered Ti6Al4V alloy and TMCs was determined by density measurement in accordance with ASTM B962 using Archimedes’ principle, and the details of the results obtained are presented in Table 2. From the table, it can be seen that the density of the sintered Ti6Al4V alloy was measured to be 4.42 g/cm³ while the sintered TMCs showed a slight reduction in the measured density from 4.36 g/cm³ to 4.27 g/cm³ and 4.27 g/cm³ to 4.16 g/cm³ as the h-BN reinforcement increased from 1 wt. % to 3 wt. % and 3 wt. % to 5 wt. %, respectively, primarily due to the relatively low density of h-BN (2.3 g/cm³) [9]. Meanwhile, it is evident that high relative densities ranging between 98.34 % and 99.77 % were attained for the sintered Ti6Al4V alloy and TMCs under the same sintering conditions.

Table 2. Details of the density measurement results.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Measured density (g/cm³)</th>
<th>Theoretical density (g/cm³)</th>
<th>Relative density (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti6Al4V</td>
<td>4.42</td>
<td>4.43</td>
<td>99.77</td>
</tr>
<tr>
<td>Ti6Al4V-1h-BN</td>
<td>4.36</td>
<td>4.39</td>
<td>99.32</td>
</tr>
<tr>
<td>Ti6Al4V-3h-BN</td>
<td>4.27</td>
<td>4.31</td>
<td>99.07</td>
</tr>
<tr>
<td>Ti6Al4V-5h-BN</td>
<td>4.16</td>
<td>4.23</td>
<td>98.34</td>
</tr>
</tbody>
</table>
Moreover, the unreinforced Ti6Al4V alloy showed near full theoretical densification (99.77 %), indicating that the SPS technique enabled effective consolidation of the alloy as a result of adequate diffusional mass transport resulting in strongly bonded particles with minor inclusion of porosity as evidenced in the SEI in Fig. 2(a) [16]. On the other hand, with the addition of h-BN reinforcement to the titanium alloy, the TMCs exhibited a progressive reduction in relative densities from 99.07 % to 98.34 % as the reinforcement content increased from 1 wt. % to 5 wt. %. The observed depreciation in densification is probably due to the inclusion of porosities (major causes of poor densification in sintered materials) and the formation of h-BN clusters at the grain boundary regions as revealed in the SEIs in Fig. 2(b-c) and Fig. 2(d), respectively. This arguably produced weak interfacial bonding within the TMCs instigated by insufficient diffusion at the matrix-reinforcement interfaces, leading to thermal mismatch between the Ti6Al4V alloy matrix and h-BN reinforcement particles [17-18].

3.4 Nanomechanical properties of sintered composites

To determine the influence of varied contents (1, 3 and 5 wt. %) of hexagonal boron nitride on the nanomechanical behaviour of the sintered TMCs, the samples were subjected to constant indentation load of 200 mN. The nanoindentation load-displacement graphs, penetration depth-time graphs of the titanium alloy matrix and TMCs were generated as shown in Fig. 4(a) and 4(b) and further analysed to obtain the measured values of nanoindentation hardness and reduced modulus of elasticity as presented in Fig. 4(c) and 4(d), respectively. In Fig. 4(a), a comparable loading and unloading trends that is smooth without any pop-in effects can be observed for all the tested samples. The region between the loading-unloading curves of Ti6Al4V alloy matrix appears wide with largest displacement which suggests that the alloy’s resistance to plastic deformation is low, which is not unusual because of the ductility of the Ti6Al4V alloy matrix [3]. Additionally, the alloy matrix displayed the highest penetration depth as shown in Fig. 4(b). This implies that the indenter’s tip penetrates the alloy matrix with greater ease compared to the reinforced TMCs. It is recognized that the extent of penetration depth has correlation with hardness of the material [3,14]. Thus, the least nanoindentation hardness and modulus of elasticity values of 5.838 ± 0.82 GPa and 115.07 ± 6.32 GPa were measured for the alloy matrix (see Fig. 4(c)).

On the other hand, it can be noticed in Fig. 4(a) that the regions between the loading-unloading curves of the reinforced TMCs appear narrow with a major shift towards the left which indicates that the reinforcements effectively minimize the displacements owing to the load sharing of the TMCs [21]. Meanwhile, the penetration depth of the reinforced TMCs as shown in Fig. 4(b) reduced drastically with time and increasing h-BN content from 1 wt. % to 5 wt. %, implying a reduced plastic deformation behaviour with increasing h-BN content.

In Fig. 4(c), it is shown that there is a continuing enhancement in nanohardness (50.66 ± 2.25-70.78 ± 3.34 GPa) and modulus of elasticity (238.69 ± 12.25-356.76 ± 21.34 GPa) with increasing h-BN content believed to be due to load transfer mechanism from the alloy matrix to the harder h-BN reinforcing particles and the effect of dispersion strengthening brought about by increased resistance to dislocation motion by the dispersed phase particles of h-BN, especially at higher reinforcement contents [12,14].
Fig. 4. Comparison of (a) Load-displacement curves (b) Penetration depth-time graph and (c) Hardness and modulus of elasticity values for sintered Ti6Al4V alloy and TMCs with increasing content of \( h \)-BN.
3.5 Tribological properties of sintered composites

The hardness behaviour of materials has been reported to have an important effect on the tribologique properties [22]. In order to assess the influence of varied contents (1, 3 and 5 wt. %) of hexagonal boron nitride on the tribological properties of the sintered TMCs, the changes in the coefficient of friction (COF) and specific wear rates of the TMCs were studied under the applied normal loads of 5 N, 10 N and 15 N. The comparison between the COF signals, the average COF against load and specific wear rates of the unreinforced alloy matrix and the reinforced TMCs are presented in Fig. 5(a), 5(b) and 5(c), respectively.

From Fig. 5(a), varying magnitudes of COF signals can be observed at the running-in period (initial stage) for all the samples. However, the unreinforced alloy matrix showed the highest magnitude of COF signal and relatively short running-in period probably due to absence of reinforcement [23]. On reaching the steady-state period, the samples showed more stable COF signal ranging between 0.02 and 0.48.

According to Fig. 5(b), the average COFs of all the TMCs can be seen with lower values when compared with the unreinforced alloy as the applied load increased from 5 N to 15 N. The TMC reinforced with 3 wt. % h-BN is found with extremely low average COF of 0.08 ± 0.011 under the applied load of 5 N. This shows the lubricity of the TMC attributed to the smooth and lubricious h-BN content [24]. It can be implied that COF is connected to the basic interaction of asperities between the counter bodies, which also explains why COF signals with sliding time are not the same for all the materials. Besides, the nanoparticles of h-BN reinforcement in the TMCs gave substantial support to the applied load which aided to reduce the point of contact between the ball and the counter bodies, thereby minimizing the average COF and excessive scratching on the materials’ surfaces [25].

In Fig. 5(c), it is shown that the specific wear rates mostly increased linearly as the applied load increased for all the materials. Meanwhile, the specific wear rate of the unreinforced alloy at each applied load: 5 N (3.391 ± 0.141 x 10^{-4} mm³/Nm), 10 N (7.272 ± 0.32 x 10^{-4} mm³/Nm) and 15 N (8.25 ± 0.304 x 10^{-4} mm³/Nm), was higher than those of the TMCs attributed to the relatively low nanoindentation hardness value of the alloy matrix as shown in Fig. 4(c) [26]. It is equally worthy to note that softer materials tend to wear more than the harder ones under the same frictional conditions [27].
Fig. 5. Comparison between (a) variations of COF under 10N load (b) average COF against load (c) specific wear rate values obtained for sintered Ti6Al4V alloy and TMCs with increasing content of h-BN.
The typical SEIs in Fig. 6 revealed the mechanism of wear and wear debris morphologies after the experiment. The unreinforced alloy matrix exhibited a characteristic abrasive wear due to plastic deformation and cutting of the ductile alloy at the microscale and adhesive wear aggravated by extensive rubbing without lubrication effect [28]. With further sliding, frictional force increased generating much heat resulting in work-hardening and welding of the wear debris on the wear track of the matrix as shown in Fig. 6(a) [29]. For all the reinforced TMCs (see Fig. 6(b-d)), wear was reduced and excessive scratching of the alloy matrix during sliding was prevented, hence, less wear debris were removed. Therefore, the wear resistance of the reinforced TMCs were significantly enhanced compared with the Ti6Al4V alloy matrix and the 5 wt. % reinforcement exhibited the greatest resistance to wear, closely followed by the 3 wt. % reinforcement attributed to high nanohardness and good lubricity [30].

![Fig. 6. Typical SEIs of the worn surfaces obtained for: (a) Ti6Al4V alloy (b) Ti6Al4V-1h-BN (c) Ti6Al4V-3h-BN and (d) Ti6Al4V-5h-BN under an applied load of 10N](image)

### 4 Conclusion

The influence of varied contents (1, 3 and 5 wt. %) of h-BN on the nanomechanical and tribological properties of Ti6Al4V matrix composites developed by SPS has been studied in this work. The following conclusions were drawn from the study:

1. SPS enabled accomplishment of well-refined grains, high density samples and development of solid matrix-reinforcement interfacial bond.
2. Evolution of secondary phases of BN, TiB and AlN played a key role in the ensuing enhanced properties of the TMCs.
3. Addition of h-BN reinforcement to the Ti6Al4V alloy matrix caused a progressive reduction in relative densities of the TMCs from 99.07 % to 98.34 % as the reinforcement content increased from 1 wt. % to 5 wt. %. The deterioration in
Continuing improvement in nanoindentation hardness (50.66 ± 2.25-70.78 ± 3.34 GPa) and modulus of elasticity (238.69 ± 12.25-356.76 ± 21.34 GPa) with increasing h-BN content were due to load transfer from the matrix to the harder h-BN reinforcing particles and dispersion strengthening effect.

5. The wear resistance of the reinforced TMCs was significantly enhanced compared with the Ti6Al4V alloy matrix. The TMC with 5 wt. % of h-BN exhibited the greatest resistance to wear, followed by the TMC with 3 wt. % of h-BN, and this was attributed to high nanohardness and good lubricity.

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