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THE IMPORTANCE OF SUBSTRATE MATERIAL IN HIGH TEMPERATURE TRIBOLOGICAL TESTING OF PDV COATINGS – A CASE STUDY

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Abstract: *Choosing the right material as a substrate for coating deposition is an important task when conducting tribological testing. The substrate must possess the appropriate mechanical, thermal and chemical properties to withstand tribological testing and to closely simulate real operating conditions. Poor material selection can lead to premature failure of the coating due to deterioration or breakdown of the substrate. Additionally, the changes of the substrate's properties at high temperatures can alter testing conditions and consequently influence the test results and create a false representation of coating's behaviour. In this study, two coatings were deposited using industrial magnetron sputtering unit, nanolayer TiAlN/TiSiN (nl-TiAlSiN) coating and multilayer TiAlN/TiSiN/nl-TiAlSiN. The former coating was deposited on three substrates: hot-work tool steel, plasma nitrided hot-work tool steel, and cemented carbide, while the latter was deposited on heat resistant stainless steel and IN-100 superalloy. The tribological tests were performed at room temperature, 400, 500, 600, 700, and 800 °C using a high temperature pin-on-disk tribometer.* Al_2O_3 *ball was used as a counter-body. After the tribo-tests, wear tracks were examined using several microscopy and spectroscopy techniques. Both coatings on all substrates displayed similar wear mechanisms at room temperature. However, at higher temperatures the failure of the substrate materials was observed. This changed the wear mechanism from abrasive to oxidative and diffusion wear and compromised the load-bearing capacity of the substrate which resulted in fracture of the coatings. As a result, the wear rate calculation was obstructed or made unreliable and, in some cases, the coating was prematurely destroyed. Additionally, the coating on IN-100 substrate displayed exceptional behaviour among the investigated combinations, even at high temperatures, due to materials microstructure. Therefore, it is concluded that the choice of substrate material has a huge influence on the behaviour and duration of the coating and should be carefully considered.*

Keywords: *TiAlSiN, PVD coatings, High temperature, Tribology, Wear rate, Substrate material*

1. INTRODUCTION

The choice of substrate material or substrate pretreatment is of crucial importance when performing tribological testing of the coatings, especially at high temperatures. Choosing the right material is not an easy task, it must take into account mechanical loads, thermal and chemical effects, adhesion of coating to substrate, etc. Apart from that, the substrate material must be chosen in a way that it can simulate behaviour of a material in real working conditions. Inadequate material selection can influence the results of tribological testing of the coating and subsequently create inaccurate portrayal of the coating's performance. On the other hand, the substrate material can sometimes be a limiting factor when testing coating's capabilities. In some cases, substrate failure can happen even before the full coating's potential has been discovered. Therefore, in this study, four different materials and one substrate pretreatment were used for subsequent coating deposition and tribo testing.

The nanostructured TiAlSiN is a contemporary coating that has recently been extensively investigated and makes a promising solution for industrial application. This coating is characterized with high hardness, fracture toughness, thermal stability and oxidation resistance [1–3]. Therefore, it was chosen in two variations for the comparison of different substrates and pretreatments.

2. MATERIALS AND METHODS

The substrate materials used in this study were hot-work tool steel (H11) (EN X37CrMoV5-1), plasma nitrided hot-work tool steel (PN H11), cemented carbide (CC), heat resistant steel (HRS) (EN X15CrNiSi25-22) and superalloy (IN-100). Nanolayer TiAlN/TiSiN (nl-TiAlSiN) coating was deposited on the first three substrates, while multilayer TiAlN/TiSiN/nl-TiAlSiN (ml-TiAlSiN) was deposited on the remaining substrates. Both coatings were deposited using two TiAl and two TiSi targets in an industrial DC magnetron sputtering unit (CC800/9, CemeCon). The ball cratering method revealed coating thicknesses of 3.6 µm and 6.8 µm for the nanolayer and multilayer coating, respectively.

The hardness of the coatings was determined using an instrumented indentation (H100C, Fischerscope), with a load of 50 mN. The standard HRC tests and Vickers hardness tester using a load of 2 and 5 kg (HV2, HV5) were used to measure the hardness of the substrates. The tribological tests of the coatings were performed at room temperature (RT), 400, 500, 600, 700, and 800 °C using a high temperature pin-on-disk tribometer. The coatings were tested against alumina counterballs with a diameter of 6 mm. A dead load of 5 N and 1 N was used to test nanolayer and multilayer coating, respectively. Linear velocity was 5 cm/s, and the number of cycles was 2000. The wear rate was calculated using the following equation: $K = V / (w \cdot s)$, where V is the worn volume (mm^3), w is the normal load (N), and s is the sliding distance (m).

After the tribological tests, the wear tracks were examined using confocal microscopy (Axio CSM700, Zeiss), 2D tactile profilometry (Talysurf, Taylor Hobson), scanning electron microscopy (SEM) (Quanta 650 ESEM, FEI) equipped with energy dispersive spectroscopy (EDS), and focused ion beam (FIB).

3. RESULTS AND DISCUSSION

The hardness of both as-deposited coatings, and the hardness of nl-TiAlSiN coating at room temperature after the tribological tests at 700 °C, is presented in Table 1. The ml-TiAlSiN coating exhibited higher as-deposited hardness. After the annealing at 700 °C, nl-TiAlSiN coating retained its hardness.

Table 1. Hardness of the coatings

Coating	As deposited HV0.005 (GPa)	Annealed at 700 °C HV0.005 (GPa)
nl-TiAlSiN	2400 (23.5)	2250 (22.1)
ml-TiAlSiN	3300 (32.4)	-

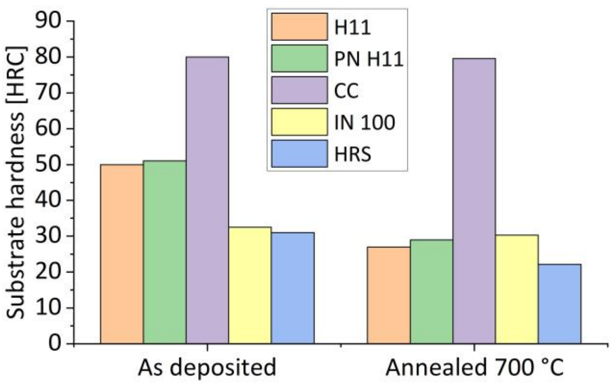

Figure 1. Substrate hardness in different conditions

The hardness of the substrates after the coating deposition, and at room temperature after the tribological test at 700 °C, is shown in Figure 1.

Cemented carbide and superalloy retained their hardness, while the hardness for remaining substrates decreased for about 20-50%. Hardness directly at high temperatures (hot hardness) was not measured. However, significant decrease of hot hardness is expected for all investigated substrates [4,5].

Representative CFM images of the wear tracks after tribo-test at different temperatures, for both coatings and all substrates, are presented in Figure 2. The wear tracks for all tests at RT are characterized by smooth regions, indicating abrasive wear mechanism. On the other hand, the wear tracks for ml-TiAlSiN coating also have adhered material present inside, which indicates an additional adhesive wear mechanism.

After the tests at 400 and 500 °C the wear tracks for nl-TiAlSiN coating are narrower than at RT, which indicates a lower wear rate at high temperatures. For tests at 600 °C, bright areas inside the wear track for the coating on H11 substrate can be attributed to pronounced oxidation of substrate material, while the wear tracks for coating on other two substrates are without bright areas. Additionally, the cracks inside the wear track were observed for the coating of H11 substrate, and their intensity increased with an increase in testing temperature.

After tribo-test at 700 °C, the wear tracks for the coating on H11 and PN H11 substrates were filled with oxide. In these cases, the coating inside the wear track is destroyed and cannot be used in these conditions. On the other hand, the coating on CC substrate does not show such damage. However, when the coating on CC substrate was tested at 800 °C, it was found that the oxidation of CC substrate occurred, and the coating was destroyed inside and outside the wear track.

As for the ml-TiAlSiN coating, the wear tracks for the coating on the IN100 substrate were slightly narrower than on the HRS substrate, except for tribo-test at 600 °C. This indicates lower wear of the coating on IN100 substrate, especially at 800 °C. The wear tracks at high temperatures exhibited a combination of abrasive, adhesive and oxidative wear mechanisms. Additionally, the coating on the IN100 substrate is characterized by thin needle-like protrusions that are randomly distributed across its surface. The worn material builds up on these surface features.

Surface of the coating on HRS substrate is smoother than on IN100 substrate, without protrusions. Additionally, the cracks can be observed inside the wear track of the coating on HRS substrate tested at 800 ° C. However, no clear signs of pronounced oxidation damage are visible inside the wear tracks of the coating on these two substrates.

A more detailed analysis of the wear tracks was done using SEM and EDS techniques. First, the surface of the wear tracks after high temperature tribo-tests was analysed. The presence of oxygen was revealed for all high temperature tests. Usually, Al-O, Ti-O and Si-O are reported to form on top of the TiAlSiN coating after being exposed to high temperatures [6].

Second, the cracks observed for nl-TiAlSiN coating on H11 substrate were analysed. SEM image of coating's cross-section prepared using FIB, after tribo-test at 600 °C, is presented in Figure 3. The cross-section was prepared across two cracks. The cracks propagate between the coating's columns and extend through the entire coating's thickness. At the coating-substrate interface a difference in height, like a step, can be seen. These cracks in the coating formed due to deformation of the substrate material (loss of load bearing capacity), at high temperatures and high contact pressure. This led to failure of the coating, since the EDS analysis revealed the presence of high amount of Fe and O inside the wear track (Table 2, Spec. 1). The Fe originated from substrate material, and Fe-O filled the wear track through these cracks, making the coating unusable in these conditions.

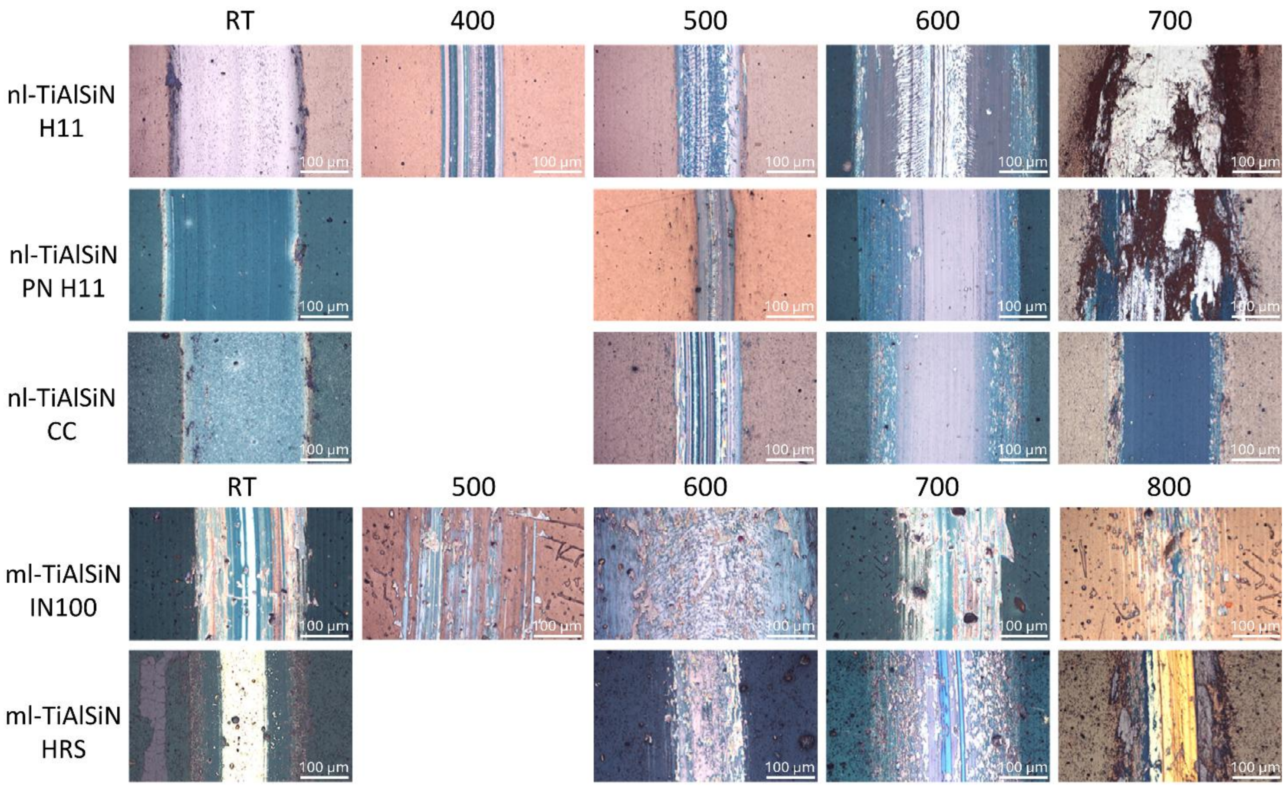

Figure 2. CFM images of the wear tracks obtained from tribo-tests at different temperatures

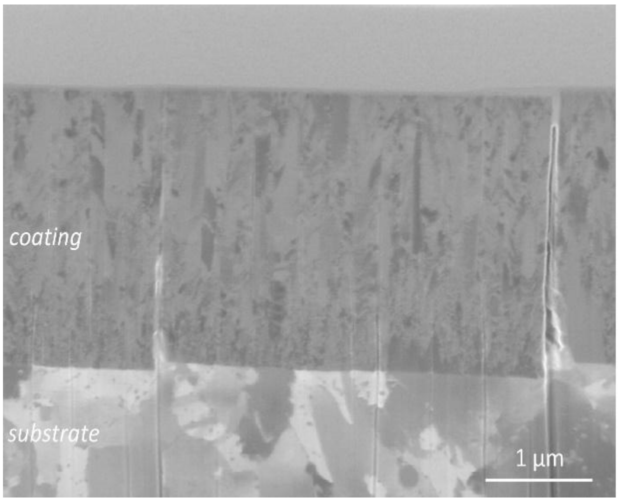

Figure 3. SEM image of FIB cross-section of the coating on H11 substrate tested at 600 °C

Next, the EDS analysis was performed after tests at 700 °C for the coating deposited on H11 and PN H11 substrates (Table 2, Spec. 2 and 3). It was found that the Fe-O completely filled the wear track of coating on H11 substrate, while it was slightly less pronounced for coating on PN H11 substrate. In both cases the coating was compromised and no longer usable.

A more detailed analysis of needle-like protrusions, for coating deposited on IN100 substrate, was performed by SEM. These features are randomly distributed over the whole surface of the coating, inside and outside the wear tracks. They are more durable (less prone to wear) than the rest of the coating and material worn during the tribo-tests accumulates on these protrusions (Figure 4.). Additionally, the alumina counterballs (not shown here) are significantly more worn against the coating on IN100 substrate, when compared with the investigated coatings on other substrates.

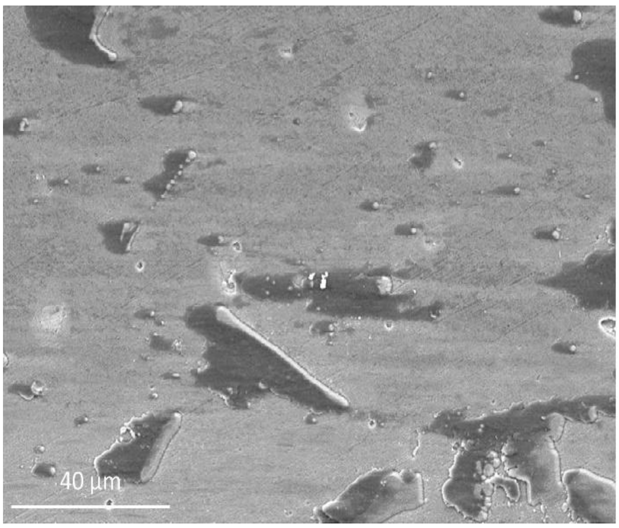

Figure 4. SEM image of ml-TiAlSiN coating deposited on IN100 substate, tested at 600 °C.

Table 2. EDS analysis performed on different locations.

Spectrum	Chemical composition [at %]					
	Ti	Al	Si	N	O	Fe
1	16.0	6.5	2.5	-	59.0	16.0
2	3.2	1.2	1.2	-	56.7	37.8
3	14.8	3.9	1.9	-	41.0	38.3
4	31.9	-	5.2	25.2	37.7	-

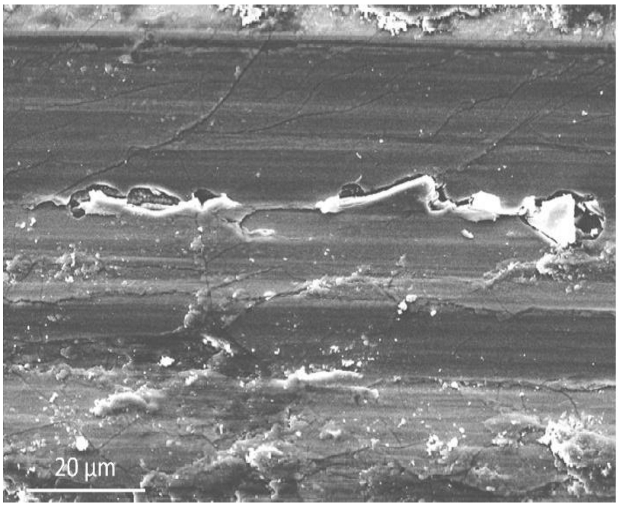

Figure 5. SEM image of ml-TiAlSiN coating deposited on HRS substate, tested at 800 °C.

Finally, the SEM image of the ml-TiAlSiN coating deposited on HRS substrate, tested at 800 °C, is presented in Figure 5. The surface of the coating inside the wear tracks is without protrusions. On the other hand, the cracks inside the wear track and delamination of the surface due to oxidation can be observed. However, even though the coating cracked, EDS analysis revealed only the presence of oxides of the coating, and not elements from the substrate (Table 2, Spec. 4). This suggests that HRS substrate along with IN100 substrate exhibited high resistance to oxidation and did not compromise the coating's integrity at high temperatures.

The wear rate of nl-TiAlSiN and ml-TiAlSiN coatings, deposited on different substrates and tested at different temperatures, is presented in Figure 6. At room temperature, the wear rate of the nl-TiAlSiN coating is comparable to similar coatings found in literature [6]. On the other hand, wear rate of ml-TiAlSiN at RT is considerably higher especially for the coating on HRS substrate, even though the ml-TiAlSiN coating has higher hardness than nl-TiAlSiN. The reason for this is the significant difference in hardness of the substrates.

Up to 600 °C, both coatings have exhibited lower wear rates than at RT, except for ml-TiAlSiN on IN100 substrate tested at 600 °C. This sample exhibited behaviour that deviated from the expected trend, therefore further and more detailed analysis is needed. The reduced wear rate at high temperatures is attributed to the formation of protective oxides within the wear tracks [6,7]. However, due to oxidation of the substrate, which caused damage of the coating inside the wear track, the wear rate of nl-TiAlSiN on H11 substrate tested at 600 °C is not relevant. For the same reason, the wear rate of nl-TiAlSiN on PN H11 substrate is also unreliable. Even though nl-TiAlSiN on CC substrate did not oxidize when tested at 700 °C, its wear rate more than doubled. The reason for this is significant decrease of hot-hardness of CC substrate at 700 °C, which resulted in its loss of load-bearing capacity [5].

The wear rate of ml-TiAlSiN coating on IN100 substrate remains low even when tested at 800 °C. The needle-like protrusions, found on the surface of the coating, significantly influenced the wear rate of this coating. These protrusions are formed by substrate carbides that were less affected by ion etching during the deposition, than the rest of the substrate's surface. The coatings typically grow epitaxially on these locations, leading to an enhanced properties at those sites [8]. When compared to the coating on IN100 substrate, the wear rate of ml-TiAlSiN coating on HRS substrate tested at 700 and 800 °C, has increased 7 and 24 times, respectively. It is postulated that the reason for considerable increase of the wear rate is decrease of hardness of HSR substrate, especially at high temperatures (hot-hardness).

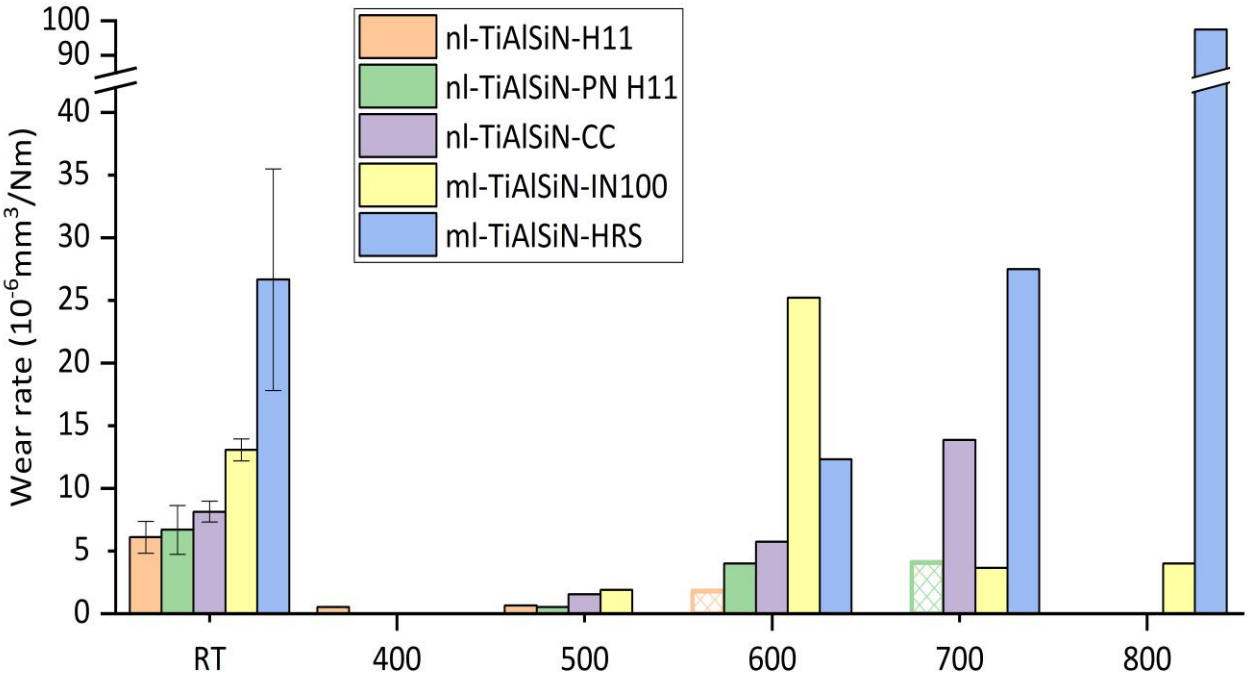

Figure 6. The wear rate of nl-TiAlSiN and ml-TiAlSiN coatings at different testing temperatures. The wear rates of hatched columns are not relevant due to oxidation.

4. CONCLUSION

In this study, nanolayer TiAlSiN and multilayer TiAlN/TiSiN/nl-TiAlSiN coatings were tribologically tested at high temperatures. From the presented results, the following conclusions have been drawn:

- After tribo-tests at 700 °C, superalloy IN100 and cemented carbide retained their hardness, while hot-work tool steel (nitrided and non-nitrided) and heat-resistant steel lost about 50% and 20% of their hardness, respectively.
- Due to formation of protective oxides both nl-TiAlSiN and ml-TiAlSiN coatings, on all substrates, exhibited lower wear rate at temperatures up to 600 °C, than at room temperature.
- Inside the wear track of nl-TiAlSiN coating deposited on hot-work tool steel intergranular cracks were observed after tribo-test at 600 °C. This resulted in premature failure of the coating, and in formation of Fe-O on the coating's surface. After tribo-test at 700 °C the substrate oxidation was even more intensive for both hot-work tool steel and plasma nitrided hot-work tool steel substrates, and it completely destroyed coatings inside the wear tracks.
- Needle-like protrusions were found on the surface of ml-TiAlSiN coating on IN100 substrate. They were more resistant to wear than the rest of the coating. This resulted in significantly lower wear rate of the coating on IN100 at high temperatures, when compared to other substrates.
- The IN100 and heat-resistant steel did not oxidize up to 800 °C. However, the wear rate of the coating on heat-resistant steel was 24 time than on IN100, due to loss of steel substrate's mechanical properties at high temperatures.

The superalloy IN100 substrate has shown exceptional behaviour at high temperatures, when compared to other investigated substrates. However, the coating on this substrate was tested using a lower dead load (1N), because of its low base hardness. Even though the formula for the wear rate calculation takes that into consideration, the contact pressures and flash temperature would still differ. Therefore, further investigation is needed using different conditions to actually confirm its advantage. Furthermore, future investigation will also include additional pretreatments of IN100 substrate with a goal of increasing its hardness.

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