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Nanoporosity improves the damage tolerance of nanostructured tantalum nitride coatings

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ABSTRACT

Conventionally, nanostructured ceramics are susceptible to brittle fracture due to processing-induced flaws such as porosity. In this study, the microstructure and deformation behavior of two nanostructured Ta_2N coatings, one with a fully-dense structure and the other with a nanoporous structure, both prepared by double cathode glow discharge technique, were investigated. It was found that the elastic modulus of the Ta_2N coatings is more sensitive to the presence of nanoporosity than their hardnesses. Compared with the fully-dense Ta_2N coating, the nanoporous Ta_2N coating exhibits a marked increase in the damage tolerance, as well as a good potential for wear-resistance.

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Nanostructured materials often exhibit superior mechanical properties compared with their more conventional coarse-grained counterparts. As such, they have attracted much scientific interest [1,2]. Despite significant advances in the development of nanocrystalline materials, there remains a lack of fundamental understanding of underlying mechanisms responsible for their processing-structure–property relationships. For example, Gleiter et al. [3] were the first to report improved ductility and superplasticity in TiO_2 nanocrystalline ceramics, mediated by grain-boundary deformation accommodation mechanisms during compressive loading. In addition, Liu et al. showed that both high hardness and toughness could be achieved by introducing a thin amorphous Si_3N_4 grain boundary phase, which increased the interfacial strength between the crystalline phase [4]. However, both simulations and experimental observations have suggested that most of nanocrystalline materials exhibited low ductility or may even be brittle. This has caused serious problems that hinder advanced structural applications of these materials [5–8]. One of the main reasons for their low ductility could be the artefacts associated with extrinsic processing, for instance the presence of pores and impurities [6]. Thus, more effort has been devoted to producing fully-dense nanostructured materials to avoid the degradation of mechanical properties caused by the presence of porosity [9]. However, conflicting results have been reported from investigations of toughness of bulk metallic glasses and nanocrystalline ceramic materials. For example, Wada et al. [10] found that the fine (20–30 μm diameter, <4% volume fraction) pores uniformly dispersed in a Pd-based bulk metallic glass (BMG) only slightly reduced the Young's modulus and yield strength, but dramatically enhanced plasticity in compression. Reddy et al. [11] also suggested that the mechanical properties of nanocrystalline B_4C ceramics, including compression strength, plasticity and toughness, could be improved by introducing nanoporosity.

Elastic modulus, E, and hardness, H, are two mechanical properties essential for the application of structural materials. The ratios between hardness and elastic modulus, i.e. H/E and H^3/E^2, have been widely quoted as reliable indicators in evaluating the plastic deformation resistance of material, especially as performance criteria that are important in defining the wear resistance of materials [12,13]. These two parameters suggest that a material with high hardness, but lower elastic modulus, possesses a higher resistance to plastic deformation. However, since it is difficult to make significant changes in elastic modulus, efforts have been made to increase the hardness of nanocrystalline coatings [14]. Additionally, the threshold load for crack initiation (P_c) in a solid material [15] is proportional to K_c^4/E^2H, which means that the damage tolerance for a material would be improved by both reducing E and H and, at the same time, increasing fracture toughness, K_c [16]. Notably, for conventional materials, high hardness is usually accompanied by high elastic modulus [17]. However, the elastic modulus of a nanostructured material is not directly correlated to its hardness, but strongly

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dependent on its grain-boundary structure and defect structure, such as porosity [18]. This provides an important basis for regulating the mechanical behavior of nanostructured materials through optimizing microstructural design.

The aim of this work is to explore the role of nanopores in controlling the mechanical properties of a Ta_2N-based coating. Both nanoporous and fully-dense Ta_2N coatings were deformed under a range of loads using a range of indentation tests. Transmission electron microscopy (TEM) and focused ion beam (FIB) were used to observe the deformation microstructures generated. Interestingly, it was found that the presence of nano-sized pores enhanced the damage-tolerance of the Ta_2N nanoceramic coating, without sacrificing its hardness. Several underlying mechanisms responsible for these improved mechanical properties are discussed.

Two Ta_2N-based nanoceramic coatings were deposited onto Ti-6Al-4V substrates using double cathode glow discharge apparatus equipped with a Ta target. Prior to deposition the surfaces of the samples were further cleaned by Ar ion bombardment at −650 V for 10 min. The process of double cathode glow discharge was described elsewhere [19]. Deposition was carried out in an Ar and N_2 atmosphere. The base pressure was 5×10^{-4} Pa and deposition gas pressure was kept constant at 35 Pa with an Ar:N_2 flux ratio of 20:1. The target/substrate distance was 10 mm; and treatment time was 1 h. For the fully-dense (FD) Ta_2N coating the deposition parameters were: target electrode bias voltage, −900 V; substrate bias voltage, −350 V; substrate temperature, 800 °C. For the nanoporous (NP) Ta_2N coating the parameters were: target electrode bias voltage, −850 V; substrate bias voltage with, −300 V; substrate temperature, 700 °C.

The crystallographic structure and residual stress of the coatings were analyzed by Bragg–Brentano X-ray diffraction (XRD, D8ADVANCE) with Cu Kα radiation. The microstructure of the coatings was characterized by scanning electron microscopy (SEM, Hitachi S3400, Japan) and field emission transmission electron microscopy (FEG-TEM, Philips CM200, Netherlands). The hardness and elastic modulus of the coatings were obtained using a nanoindentation tester (Ultra-Micro Indentation System 2000) equipped with a Berkovich indenter.

In order to gain a deeper understanding about the influence of nanoporosity on the mechanical properties of the two Ta_2N coatings, both load cycle indentation and multiple partial unloading indentation modes were used during nanoindentation testing [20]. Each multiple partial unloading indentation test was conducted with five loading/partial unloading steps. The largest applied load for each loading step increased from 80 to 400 mN. After each loading step, the indenter is unloaded to 20% of that current load before being loaded to the next load. Since the unloading process is totally elastic, the mechanical properties will not be greatly impacted by the repetitive unloading process [21,22]. Meanwhile load cycle indentation was also performed on the two coatings with a range of maximum loads from 80 mN to 400 mN, identical to the five loads described in the multiple partial unloading indentation process. For both the partial unloading and load cycle indentation, the maximum penetration depth was carefully controlled to be <10% of the coating thickness to avoid any influence from the substrate [23]. In addition, 1000 g indents were applied to the two Ta_2N coatings using a microindentation system equipped with a Vickers indenter. These experiments were performed to qualitatively evaluate the damage tolerance of the two Ta_2N coatings. The cross-sectional morphology of the indentation sites were examined through TEM samples sliced from the center of the 1000 g indent on the nanoporous Ta_2N coating using focused ion beam microscopy (FIB, FEI xP200, FEI).

As shown in Fig. S1 (in the supplemental material), the XRD patterns of both coatings show only characteristics peaks for the hexagonal structured Ta_2N phase, which matches well with JCPDS No. 26-0985. The calculated residual stress of the fully-dense Ta_2N coating (-1.65 ± 0.22 GPa) is slightly larger than that of NP-Ta_2N coating (-1.25 ± 0.19 GPa). An overwhelmingly strong peak arising from the (101) reflections suggests that the two Ta_2N coatings grew predominantly with a (101) preferred orientation. From the cross-section SEM images (Fig. S2(a) and (b) in the Supplemental material) it can be seen that both two Ta_2N coatings display a dense microstructure free from visible pores or cavities with a uniform thickness of ~20 μm. It appears that both coatings are well bonded to their substrates.

Fig. 1 shows typical cross-sectional bright-field TEM images taken from the two Ta_2N coatings, together with the corresponding selected area electron diffraction (SAED) patterns. TEM observations suggest that both coatings exhibit a homogeneous microstructure, comprised of approximately equiaxed grains with a typical grain size ranging from 15 to 20 nm [24]. An intense Ta_2N (101) ring in the inset selected area diffraction patterns shown in Fig. 1(a) and (b) provides further evidence that both Ta_2N coatings have a strong (101) texture, in agreement with the XRD data. On closer inspection, it can be found that no visible porosity can be observed in the fully-dense Ta_2N coating, whereas a large number of nanopores, with an average diameter of 2 nm, are uniformly distributed at the grain boundaries and triple junctions for the nanoporous Ta_2N coating. According to Fig. 1(b) the volume fraction of porosity in nanoporous Ta_2N coating can be calculated as ~11%. This structural transition from the fully-dense to the porous structure for the two as-deposited Ta_2N coatings is intimately associated with the different deposition parameters. Compared with the nanoporous Ta_2N coating, the fully-dense Ta_2N coating is prepared at a higher deposition temperature combined with increased ion bombardment induced by an increased substrate negative bias. The higher deposition temperature increases the surface mobility of adatoms, allowing the adatoms to diffuse to more equilibrium positions and to overcome any self-shadowing effects exerted by the previously deposited atoms. Meanwhile, the increased ion bombardment through raising the negative bias of the substrate enhances the nucleation density by forming nucleation sites and also decreases the incidence of interfacial voids [25]. Remarkably, the difference in deposition temperature has little influence on the grain sizes of the two Ta_2N coatings.

Fig. 2(a) and (b) shows typical load–displacement curves for the two Ta_2N coatings aquired from both multiple partial unloading and load cycle indentation tests. All the curves exhibit displacement continuity at each loading and unloading step without any detectable pop-in or pop-out events. It should be noted that the maximum indentation depth is far <10% of the coating thickness, suggesting that any contribution to the measured mechanical properties from the substrate is likely to be negligible. As shown in Fig. 2(a), for the fully-dense Ta_2N coating, the two curves determined by the two loading modes almost merge with each other. On the contrary, in the case of the nanoporous Ta_2N coating (Fig. 2(b)), at the low loads, the multiple partial unloading curves almost overlap with the load cycle indentation curves; however, with increasing indentation load, they diverge from the load cycle indentation curves towards a shallower penetration depth, suggesting an increased resistance to local plastic deformation.

Fig. 2(c) and (d) shows the comparison of hardness and elastic modulus determined by the two loading modes as the function of maximum indentation loads for the two coatings. For both loading modes, the measured hardness (H) and elastic modulus (E) for the full-dense Ta_2N coating slightly decrease with an initial rise in maximum indentation load, due to the indentation size effect [26,27] and, subsequently, reach stable values. It is obvious that, at same indentation load, the H values calculated from the multiple partial unloading curves are a little higher than those obtained from the load cycle indentation mode, whereas higher E values were obtained from the load cycle indentation curves. The reasons for the higher H and lower E in multiple partial unloading mode compared to load cycle mode can be attributed to material creep and slower unloading steps during the load cycle mode, respectively [28,29]. For the nanoporous Ta_2N coating, the variations of the H and E obtained from the load cycle indentation mode versus indentation load show a similar trend to those for the fully-dense Ta_2N coating. However, for the multiple partial unloading mode, the measured H and E values initially decrease and then increase with increasing

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