



An insight into formation of nanostructured coatings on metallic substrates by planetary ball milling

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ABSTRACT

Recently, high-energy ball milling has been shown to be a potential room temperature method for applying a coating to substrates inside the milling vial. The experiments discussed in this paper were performed in the hope of broadening the applicability of the technique to different types of coating materials and to different substrate positions. Ni and SiC powders were separately employed as the coating material to be mechanically deposited on an Al substrate fixed at either the top end or the bottom end of the milling vial. The obtained coatings were characterized using X-ray diffraction, scanning and transmission electron microscopy. Mechanical properties of the coatings, including wear performance, cohesion and bonding strength, were also studied. Under the milling condition used in the present study, SiC coatings showed low microhardness and wear resistance irrespective of the substrate position, mostly due to an insufficient degree of consolidation. However, excellent results were obtained for Ni coatings produced under similar milling condition. It was realized that the top position for the substrate provided a remarkably better microhardness, wear resistance and adhesion strength. Ni coating on the top substrate with an average crystallite size of about 58 nm, showed a microhardness of 422 ± 12 HV that is at least 30% higher than that of a typical electroplated Ni coating. The difference in coating characteristics for the two substrate positions was attributed to different deposition mechanisms.

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1. Introduction

Nanostructured coatings have received great attention due to their superior characteristics compared to their microstructured counterparts. Decreasing the grain size of the coating to the nanometric level leads to a higher resistance to deformation and initiation of crack and consequently development of super-hard and super-tough wear-resistant coatings [1]. Nevertheless, there are still great challenges for the development of many nanostructured coating/substrate systems, e.g., achieving an appropriate adhesion between the coating and the substrate which generally requires high deposition temperatures. However, at these temperatures, it is quite difficult and even impossible to hinder the growth of nano crystallites in the coatings. Oxidation of either coating or substrate material is another limitation of high temperature deposition methods which severely restricts their applicability.

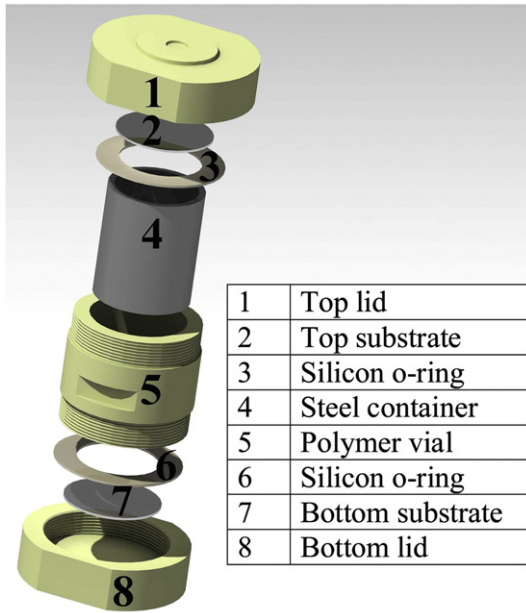
Recently, combination of mechanical alloying and surface ball milling processes has been widely used for the mechanical coating (MC) of metallic substrates. One of the most important advantages of

the MC process is enhancement in coating/substrate bonding due to mechanical activation of sample surfaces, which could be easily fulfilled at ambient temperature and atmospheric pressure [1,2].

Researches in the field of MC could be classified from different viewpoints: (i) the equipment used, (ii) the coating material and (iii) the substrate position. One of the most common tools for MC is planetary ball mills. They have been largely used for the deposition of various metallic [3–5] and inter-metallic [6–8] coatings. Milling balls [9–11], sidewall of the container [12–14] and a small sample loaded into the vial [6,15] have been investigated as different target substrates. However, there is no report on the deposition of a non-metallic coating via planetary ball milling. Yet mechano-reactor [16] and ultrasonic-based vibration chambers [17,18] and also SPEX 8000 mixer mill [19] have been actually employed for applying non-metallic coatings such as TiN, WO₃ and hydroxyapatite on metallic substrates. Moreover, no attempt has been made to deposit coatings of any type on the substrates placed at the bottom end or the top end of the vial of planetary ball mills. This is probably due to the deficient understanding of the ball trajectories in real planetary ball milling, which is rooted in modeling and simulation studies.

Modeling ball trajectory in the planetary ball mill has been the subject of many investigations (e.g., 20, 21). Neglecting the gravity force applying to the ball, two types of forces are considered: centrifugal forces towards the center of the sun-disc and milling container and

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1	Top lid
2	Top substrate
3	Silicon o-ring
4	Steel container
5	Polymer vial
6	Silicon o-ring
7	Bottom substrate
8	Bottom lid

Fig. 1. A schematic illustration of the setup used in this research.

frictional forces originating from the interactions between the ball and the milling container. Based on the ratio of vial to disk angular velocity called transmission ratio ($T_r = -\omega_v/\omega_d$), three different types of trajectory could occur while milling: (i) chaotic for $|T_r| < 1$, (ii) impact + friction for $1 < |T_r| < 3$ and (iii) friction for $|T_r| > 3$ [20]. Impact + friction mode is the one in which planetary ball mills work that leads to the cascading movement of the balls and powder. Chattopadhyay et al. [22] proved that the sign of tangential component of the impact force (F_t) changes from positive to negative at $T_r = 1$. In the case of positive F_t , the ball and the vial rotate in the reverse direction during an impact, which would tend to sweep the entrapped powder out of the impact zone. Consequently, direct contact between bare ball and vial surfaces would lead to the higher level of wear and contamination. On the other hand, negative F_t retains the powder particles in the impact area, enhancing effective deformation of entrapped powder. As the vial rotates, the force on the ball is oriented towards the exterior of the mill, leading to the sticking of the ball to the vial wall. At a certain point, called the takeoff point [21], the forces on the ball change direction, throwing it across the vial. Subsequently, the linear movement of the ball eventually leads to a collision with the vial wall. At this point, the ball is considered to stick to the wall without rebounding and then follow the movement of the vial until it again reaches the takeoff point. It is generally agreed that the balls in the mill are lifted up and then thrown away with a certain velocity to collide with the vial wall. Noteworthy, no specific statement has been made on the ball collision to the top or bottom ends of the vial, which is most probably due to the fact that all models have been derived based on a single ball or a limited number of balls. Rationally, the presence of a large number of balls can disturb the well-defined cascading ball trajectory predicted by the models, as the mean free path of a flying ball is shortened due to its successive collisions with the other moving balls. It therefore creates such a chaotic ball movements in the vial. The present authors believe that any spot of the inner surfaces of the vial of a planetary ball mill (be it the side, top or bottom walls) has a good chance to be impacted by the flying balls provided a large number of balls is employed.

The investigation reported in this paper was aimed at studying the formation and evaluation of the coatings on Al substrates fixed at either of the two ends of the vial of a planetary ball mill. The advantage of these two positions compared to the side wall position or a free-flying specimen arrangement is the possibility of coating flat substrates without any deformation or dimensional changes. Ni powder alone or SiC

powder alone was employed as the coating material to compare the development and properties of a ductile and a brittle coating that is built when using a planetary ball mill.

2. Experimental

A specific setup for the milling vial was designed and fabricated as illustrated in Fig. 1. The main body of the vial was made of a wear-resistant polymer in which a stainless steel tube (internal diameter of 50 mm) was inserted. Commercially pure Al substrates, in the form of circular plates of 2 mm in thickness, with a microhardness of 85 HV_{0.05} and an average roughness (Ra) of 0.5 μm , were fixed at either the top end or the bottom end of the vial by means of similar screw lids. Before the coating process, the surface of each substrate was cleaned ultrasonically. As-received Ni (Inco, Canada, >99.5%, ~10 μm , cubic, spherical shape) or SiC powder (domestic, >99.5%, ~10 μm , trigonal, irregular shape) was used as the coating material. The MC trials was carried out in a planetary ball mill (VSTW-PM2, Iran) operated at a fixed sun-disc rotation speed of 300 rpm for 5 h. In all coating treatments, half of the vial was filled with equal number of 6 and 8 mm diameter bearing steel balls. The ball-to-powder weight ratio for all coating trials was 30:1. An additional 20-h coating trial was also carried out for deposition of SiC coating on two substrate positions in order to investigate the effect of the increased milling time.

The phase composition of the coatings was studied by X-ray diffraction (XRD) analysis using a PANalytical, X'Pert Pro MPD with a Cu $K\alpha_1:K\alpha_2$ radiation ($\lambda = 1.5418 \text{ \AA}$). XRD patterns were labeled according to the JCPDS file number of 04-0787 and 04-0850 for Al and Ni phases, respectively. Topas program was used for Rietveld refinement of XRD patterns. The surface morphology and cross-sectional microstructure of the coatings were studied using a VEGA II TESCAN scanning electron microscope (SEM) equipped with energy dispersive X-ray spectroscopy (EDS). Transmission electron microscopy (TEM) was also carried out using a Philips CM120 with an operating voltage of 120 kV. Microhardness were measured using an MHV-1000Z digital Micro Vickers hardness tester at a load of 50 g and a dwell time of 15 s. Eight measurements were carried out on each sample and the results were averaged. Dry sliding pin-on-disc wear tests were performed in a laboratory atmosphere at

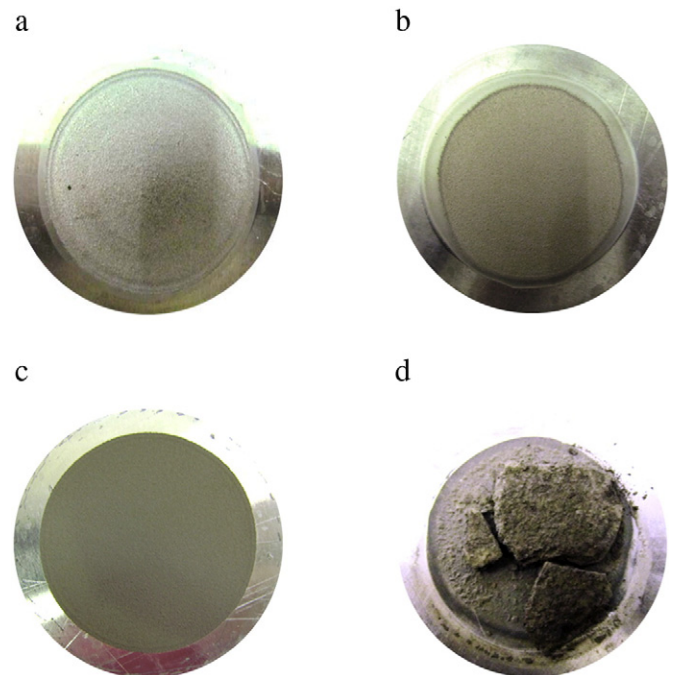


Fig. 2. Photographs of (a) Ni-T, (b) Ni-B, (c) SiC-T and (d) SiC-B coatings. The diameter of plate is 45 mm.

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