Influence of target power and temperature on roughness and tribology of vanadium-carbon based coatings

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Abstract. In the manufacturing of polyethylene terephthalate bottles, steel molds are used for the blowing process, these molds must have low roughness and friction coefficient as well as mirror-like aspect. This impacts final product features such as transparency and demolding. To achieve this, it is necessary to polish the mold cavities using abrasives such as diamond paste or alumina in suspension; however, no further protection against corrosion and wear can be achieved along its use. In this sense, physical vapor deposition coatings, such as balanced magnetron sputtering, are used to this aim giving average roughness values of 0.05 µm, along with superior corrosion and tribological behavior. Vanadium and Carbon coatings were deposited on American Iron and Steel Institute AISI 1045 steel, which is used in the manufacture of polyethylene terephthalate bottle molds, by means of sputtering technique with balanced magnetron. Deposition experiments were carried out in 8 samples during 30 minutes in an argon atmosphere using 2 to the 3-factorial design with different values of power applied to Vanadium and Carbon target and deposition temperatures. Tribological and roughness behavior was studied in order to evaluate the effect of power applied to target on those properties. In all samples low deposition temperature main effect leads to reduced roughness, in the same way, that low power applied to Vanadium target (40 Watts); however, that roughness is reached when the higher 50 Watts are applied on Carbon target. Similarly, lower coefficients of friction are associated with low roughness and therefore to abovementioned deposition conditions. There a clear effect of low power applied to Vanadium target and temperature to maintain reduced roughness and coefficient of friction which are optimal to coat polyethylene terephthalate bottle molds.

1. Introduction
In 2018, approximately 4.3 million tons of rigid polyethylene terephthalate (PET) packaging were placed on the European market of which approx. 79% were bottles and the remainder trays [1], this data gives an idea of the immense existing market for this type of bottles. Several manufacturing processes are used in its fabrication, being the most widespread, and the so-called extrusion blow molding process. In this process, molds are made of steel, which has a cavity where a preform or parison produced by injection molding is introduced, this preform has been previously heated in an infrared-halogen resistance furnace up to approximately 120 °C, subsequently, the parison is blown and stretched into the mold axially and radially, where it assumes a bottle shape [2].

Manufacturing of molds requires low values of roughness and friction coefficient as well as a mirror finish on the surface of the cavities after the machining process since this will influence the characteristics of the final product such as transparency and easy demolding. The most common practice to obtain this low roughness is to polish the mold cavities with abrasives such as diamond paste or alumina suspension. Applying this practice, optimal roughness and mirror finishing characteristics can
be obtained; however, it is a practice that requires a lot of time and skilled operators since it is almost handmade. In addition, since it is carried out directly on the substrate, it does not protect the cavity from corrosion or wear during its use [2].

For this reason, coatings arise as an alternative to mold protection against wear and corrosion mainly. At an industrial level, physical vapor deposition (PVD) is extensively used for this aim. In fact, balanced magnetron sputtering PVD technique is one of the most interesting due to its coatings present a very low level of roughness (approximately 0.05 Ra µm) [3], excellent mechanical and tribological properties and good adhesion [4]. In this way, a series of vanadium carbide (VC) coatings were synthesized by the sputtering method, with different power densities in the vanadium (V) and carbon (C) targets and different temperatures in the sputtering chamber with the aim of determinate their influence over roughness and tribological behavior [5].

2. Methodology
The deposition parameters and the instruments used to obtain the different properties of the coatings are shown below.

2.1. Coating deposition
In order to evaluate the factors that control the value of a deposition parameter a design of experiments (DoE) was used. Dispositions experiments were done in 8 previously mechanized samples for 30 minutes in an argon atmosphere using 2 to the 3-factorial design with different values of power applied to V and C target and deposition temperatures. Variations of the power parameters were made with values of 30 W/cm² and 90 W/cm² on the vanadium and carbon targets, meanwhile sputtering temperature values were of 18 °C and 200 °C. Average rotation of the substrates, deposition time of 30 minutes and argon flow of 30 cm³ were selected as fixed parameter; Table 1 shows the PVD coating parameters.

| Table 1. Design of experiment for coating deposition. |
|-----------------------------------|----------|----------|----------|----------|----------|----------|
| Variable parameters         | Low value | High value | Sample   | Power V (W) | Power C (W) | Temperature (ºC) |
| Power V (W)            | 40        | 90        | 1        | 40         | 30         | 18        |
| Power C (W)            | 30        | 50        | 2        | 90         | 30         | 30        |
| Temperature (ºC)       | 18        | 200       | 3        | 40         | 50         |           |
|                        |           |           | 4        | 90         | 50         |           |
| Substrate rotation    | 10 rpm    |           | 5        | 40         | 30         | 200       |
| Deposition time       | 30 min    |           | 6        | 90         | 30         |           |
| Flux of argon         | 30 sccm   |           | 7        | 40         | 50         |           |
|                        |           |           | 8        | 90         | 50         |           |

2.2. Roughness measurements
The roughness measurements were carried out only in the coated side of each specimen under normal laboratory atmosphere conditions, temperature around 20 °C and relative humidity between 18% to 20%.

For these measurements, was used the portable roughness tester PCE-RT 1200 equipped with diamond stylus tip diamond radius 90 °C, twelve (12) tests were done per sample, distributed in 6 measurements in 2 different zones of the coating with the aim to assure reproducibility. Also, roughness sample without coating was measured.

2.3. Tribological measurements
The tribological characterization was developed using a Microtest MT/10/SCM ‘pin on disk’ type tribometer, the method used to make the measurements was the American Society for Testing and Materials (ASTM) G99-17 standard [6], The specimens were ultra desonic cleaning before tests. Experiments were carried out without lubricant, at a laboratory temperature of 25 °C and relative humidity of 60%. The wear parameters considered were 15 cm/s, travelled distance of 1000 meters and applied load of 10 N.
3. Results and discussion
The results obtained by varying the different parameters are discussed in the following sections.

3.1. Roughness measurements
In Table 2 are shown the roughness measurements of VC coatings with the 8 different deposition parameters. The analysis was done taking into account the 2 different deposition temperatures which were 18 °C and 200 °C and the same combinations of power to V and C targets as detailed in Table 1.

In general roughness values range from 1.333 μm to 3.649 μm; being lowest values those associated with the low temperature and power applied to V target of V, 18 °C and 50 W, respectively.

In contrast, poor surface conditions were reach when higher temperature was used in the vacuum chamber, for instance sample 6 and 8. Figure 1 depicts the relationship of roughness with temperature of deposition and power applied to carbon and vanadium targets.

| Samples | Results Ra (μm) | Standard deviation |
|---------|-----------------|-------------------|
| 1       | 2.047           | ± 0.686           |
| 2       | 1.593           | ± 0.427           |
| 3       | 1.333           | ± 0.664           |
| 4       | 2.609           | ± 0.718           |
| 5       | 2.313           | ± 0.717           |
| 6       | 3.649           | ± 1.150           |
| 7       | 1.938           | ± 0.331           |
| 8       | 2.912           | ± 0.490           |

Figure 1. Roughness performance as a function of deposition temperature and power applied to V and C targets according to DoE in Table 1.

It is clear that roughness increases with temperature regardless of the change in power in carbon and vanadium targets. In sample 1 and sample 5, according to factorial design, power on V and C targets were 40 W and 30 W, respectively and deposited at both 18 °C and 200 °C; in this case at 200 °C roughness increases 11.5% compared with roughness reach at 18 °C. In the coatings 2 and 6, the trend is similar, increases of roughness with high temperature is around 56.4%; nevertheless, in that couple the power applied to V increases to 90 W. It is important to note that in this pair of tests the difference between the powers of the vanadium and carbon targets was the highest.

Similarly, sample 3 and sample 7 are affected by higher substrate holder temperature, in this case, where the power of the carbon target is 40 W and the vanadium target is 50 W, the roughness increased by 31% when the temperature rise from 18 °C to 200 °C. Finally, roughness of sample 4 and sample 8 are similar, 2.609 μm and 2.912 μm, which implies an increase close to 10%.

Hence, it can be said that exists a relationship between roughness and temperature, that is higher surface roughness corresponds with higher vacuum chamber temperatures. This behavior may be related to the initial cluster sizes become larger with an increase in substrate temperature, most likely as a consequence of coalescence of clusters in island growth mode [7]. Surface diffusion length of clusters also increases as substrate temperature rises, which causes higher surface roughness [8].

3.2. Tribological measurements
The optical images of wear track are shown in Figure 2. The sample of Figure 2(a) to Figure 2(d) were coated at a temperature of 18 °C and those between Figure 2(f) and Figure 2(h) at 200 °C. The coating 1 (black line in Figure 3) present sustained increase in the friction coefficient ranging from 0.10 to 0.23; from a distance of 400 meters there is a tribological transition that leads to coefficient of friction close to 0.40 at the end of the test. No material detachment is observed in Figure 2(a) deducing that the wear phenomena is abrasive type, the wear area was calculated at 49.97 mm² (see Figure 2(a) to Figure 2(h)).
which is similar to the areas obtained for the odd specimens except specimen Figure 2(b), which wear area was 43.78 mm².

In sample 1, friction coefficient range between 0.10-0.30. No tribological transition is observed and therefore stabilization of the friction coefficient until the end of the test was observed. In the same way of sample 1, no material detachment was detected.

In sample 3 there is different behavior since shows the lower friction coefficient, which can be as low as 0.18 to 0.22, this behavior is explained by the greater homogeneity achieved in this coating, finding a stabilization in the friction coefficient from 100 m to 800 m. From 700 m a slight increase in the coefficient of friction is due to growth of adhesion causing oscillation of the friction values generating a mechanism known as stick-slip produced by the formation and subsequent removal of debris by indenter [9-11] Further, the area obtained was 36.56 mm², which is the lowest of all coatings tested and therefore present better wear values.

In sample 4 the friction coefficient increases from 0.15 meters to 0.40 meters until 263 meters, distance in which a tribological transition occurs, lowering the friction coefficient again to 0.20 and finally rising up to 0.40, which indicates coating surface inhomogeneity that produces stick-slip phenomenon as described in the previous sample [9,10]. Wear area value was 76.83 mm² (Figure 2), which is like sample 8 and match with higher power applied V and C targets. Sample 5 to sample 8 were deposited at the same power to C and V targets that sample 1 to sample 4, as can be seen Table 1, but using a high temperature of 200 °C in the vacuum chamber instead of 18 °C. In general, these samples present higher values of wear areas, which suggest that temperature is a critical value on tribological performance as will discussed later in section 3.3.

In this sense, sample 5 compared with sample 1 shows a similar the friction coefficient, black line vs. magenta line in Figure 3 with a friction coefficient between 0.075 and 0.35; however, wear area is 5% higher. This tendency is more accentuated when sample 2 and sample 6 were compared; since in the latter, its wear area increases up 96.92 mm², that is, 54.8% higher.

In the case of sample 7, compared with sample 3, the change is less drastic, light blue line vs. dark blue line in Figure 3; as expected wear area increases a 26% when high temperature is used. Finally, sample 8 presents one of the worst wear values, only beat by sample 6; nevertheless, no effect of temperature is observed compared with sample 4 using the same power on V and C targets. Wear area just increased by 2% when higher temperature was used.

**Figure 2.** Track area of eight coated samples (a) 49.97 mm²; (b) 43.78 mm²; (c) 36.56 mm²; (d) 76.83 mm²; (e) 52.51 mm²; (f) 96.92 mm²; (g) 49.38 mm²; (h) 78.32 mm².
3.3. Design of experiments analysis

The results shown in the two previous sections show an important influence of the temperature in the vacuum chamber and the potential applied to C and V targets on the roughness and the friction coefficient. Thus, in order to analyze in more detail, the abovementioned parameters, and taking into account the design of experiments proposed in the present work, in Figure 4 are depicted the main effects obtained using Minitab® statistical analysis software.

The statistical analysis makes it clear that the main factors affecting roughness and the wear area are the high power of vanadium target, together with the high temperature; however, power on C target seems to not affect those properties as can be seen in Figure 4(a) and Figure 4(b). With power on the vanadium target of 90 W and a deposition temperature of 200 °C, the highest values of roughness and wear area is obtained, which is not suitable for application in molds for blowing bottles.

For this reason, a contour analysis was performed to identify the appropriate vanadium power and temperature values for adjusted values of roughness and wear area, which in this case were 1 μm to 2 μm and 30 mm² to 50 mm², respectively. The results of this analysis are detailed in Figure 4(c) which shows a maximum power value in the vanadium blank of 60 W and a maximum deposition temperature of 150 °C (white area).
Hence, it can be concluded that very high values of temperature and power in the targets lead to poor surface performance which may be a consequence of coalescence of clusters in island growth mode and high energy diffusion due to temperature which also causes higher surface roughness [12]. Moreover, it is well known that the increasing power produce particles with high energy in the target and, therefore, increasing bombardment of high kinetic energy particles on the substrate, resulting in high values of surface roughness [13]. At the same time, the vibration of target particles on substrate surface due to elevated kinetic energy also induced higher surface roughness and in consequence worse behavior against wear [14].

4. Conclusions
VC coatings were deposited on AISI 1045 steel by sputtering, changes in the power applied on the carbon and vanadium targets and temperature at which the coatings were deposited were carried out in order to obtain the as low as possible values in roughness and friction coefficients to its application in the manufacturing of PET bottle blow molds. Factorial design depicts that the mains effects on roughness and friction coefficient are the power on the V targets along with deposition temperature. In general terms, it is observed that the lower the power on the targets and the temperature is close to 18°C, the lower the roughness and friction coefficient. To obtain VC coatings by sputtering, the optimal power and temperature parameters are 40 W in the vanadium target and 50 W in the carbon target at 18°C temperature, in order to obtain the best roughness and coefficient of friction properties, for its use in stretch blow molds for PET bottles manufacturing.

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