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Study of Wear Phenomena of Coatings for Die-casting

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Abstract. During high pressure die casting process, molten metal (aluminum) is injected with a die casting machine under considerable pressure into a steel mold or die to form products.

Usually, during the process, the components can undergo several damages such as hot and abrasive wear, metal corrosion and plastic deformation.

In order to increase the die life, a surface modification through the application of a Physical Vapour Deposition (PVD) coating can be done.

This paper deals with the wear behaviour of a nanocomposite coating (AlSiTiN) deposited onto hot-working tool steel and onto a ion nitrided hot-working tool steel.

The characterization includes: nanoindentation test, adhesion test, wear testing at high temperature and optical profilometry.

Results showed that the nitriding step had a beneficial effect in terms of hardness, while adhesion between the coating and the substrate was reduced .

The film exhibited good wear resistance, with adhesion phenomena as main mechanism.

Introduction

Dies for forming metal parts are very complex and stressed components due to abrasive and hot wear, thermal fatigue and plastic deformation due to the die surface exposure to the liquid aluminium, the motion of the liquid aluminium and the solidification and ejection of the casting [1,2,3].

In addition, the high temperatures may cause oxidation of the die surface.

All this damaging conditions can contribute to a decrease of die casting life, increasing the cost, due to the downtime, die replacement and repair.

In order to reduce or minimize the soldering phenomena, an effective system is to modify the die surface characteristic through coatings.

During the last decade the attention was focused on the Physical Vapour Deposition (PVD) since, compared to the Chemical Vapour Deposition (CVD) method, presents some advantages such as: low-temperature processing, use of non-toxic gases and simple deposition process.

In particular, the cathodic arc deposition have attracted more attention thanks to the high ionization, good homogeneity of the coating and the possibility to create a nanostructured coating. The nanocomposite structure are composed of at least two phases: a nanocrystalline and an amorphous phase, or two different nanocrystalline phase [4]. Because of the grain dimensions (<10nm) the dislocation inside the structure are hindered and consequently the hardness is high. The PVD coating system presents also the opportunity to create a multilayer structure. [5,6] This type of coating shows higher crack resistance, blocking cracks propagation, than the monolayer one, because of the alternating layer of different materials in nanometric scale.

Multilayers present other advantages, like the promotion of a strong adhesion between the coating and the substrate, a high wear resistance with a low chemical reactivity and low friction, the hardness and toughness increase with respect to monolayers.

The purpose of this work is the study of the high temperature wear resistance of a nanocomposite PVD coating (AlSiTiN) deposited onto H11 and ion nitrided H11. The film was characterized in terms of the adhesion by scratch test, hardness by nanoindentation test and wear resistance against aluminium pin at high temperature.

Experimental

Coating Preparation

AlSiTiN nanocomposite coating was deposited onto a H11 and H11 ion nitrided high speed steel, a HSS steel commonly used for dies.

Coating deposition was done using a physical vapour deposition (PVD) prototype unit installed at Clean NT Lab.

The machine was equipped with the Lateral Arc Rotating Cathodes (LARC[®]) system, with two cathodes. During the process, evaporated metals and metal alloys enter the plasma state to combine with the ionised process gas (nitrogen) and eventually condense on the substrate surface, as part of ceramic compounds.

Spinoidal decomposition allows to build TiN nanocrystalline structures dispersed in a Si₃N₄ amorphous matrix, with typical crystallite size of about 10 nm.

The nanocomposite coating presents the following structure: a single layer, a gradient layer, a multilayer part (5 layers) and an external layer.

The single layer is composed of TiN, which role is to enhance the adhesion between the substrate and the other part of the coating. The gradient layer makes the transition between the single layer and the multilayer part.

The multilayer is the main part of the coating and it has to support the mechanical and thermal strains. This part presents an alternate multilayer structure A-B. Different arc current values of Ti cathode 2 vs. AlSi (cathode 2) were set changing the arc current between 59 and 240% in order to create the A-B coating structure. The external layer has the same composition of the second last layer.

Laboratory Test

The coating's thickness was evaluated using a ball erosion test machine.

A 20 mm diameter steel ball and a diamond suspension 0,1 μ were used to make a crater on the coating surface. Thanks to a standard formula it's possible to measure the coating's thickness.

Vickers microhardness was determined by a FISCHERSCOPE HM 2000 XYm (Fischer, NIS-University of Turin, Turin, Italy) with WIN-HCU software. It's a computer-controlled measuring system for microhardness testing and material parameters determination, according to the standard ISO 14577.

Ten indentations were made. Each test was performed to give a cycle with the same rates on loading and unloading, using the following conditions: Peak load of 1000 mN, loading rate of 50 mN·s⁻¹.

Through a CSM Revetest equipment the coatings adhesion was measured. The machine is equipped with a acoustic emission and frictional force measurement. Scratch was performed using a 200 μm radius Rockwell C diamond stylus and the applied normal load L was continuously increased until the substrate material was exposed.

The tests were made with the following parameter: load 1-100N, load increase rate 100 N/min, penetrator's travel speed 10 mm/min. After the test, the coatings were evaluated using optical microscopy. The critical loads (L_c) allow to evidence the different behaviour of the coating delamination.

The first one (L_{c1}) represents the cohesive failure, where the first crack appears, while the second one (L_{c2}) corresponds to the complete detachment of the coating.

To test the coating's wear resistance a CSEM high-temperature tribometer, using a pin on disc configuration, was used.

The apparatus comprised a rotating shaft arrangement on which a specimen holder, made of stainless steel, was attached. A vertical loading arrangement, attached to a ball, was lowered onto the rotating specimens to produce a circular wear track.

The wear test were performed using a aluminium pin of 6 mm of diameter, with a constant load of 10 N, a linear relative speed of 25 cm/s in dry conditions. The wear tests duration was of 5,000 laps. Experiments have been run at 450°C. At the end of the tests, the wear track was measured using a profilometer Talysurf CCI 3000Å (cut-off length of 0.8 mm). Ten measurements per each sample were obtained with the aim to have a representative average value. Failure patterns of the films after the tribological test were observed by scanning electron microscope (SEM) using a ZEISS EVO 50 XVP with LaB6 source, equipped with detectors for secondary and backscattered electrons collection and EDS probe for elemental analyses.

Results

The coating thicknesses, the surface roughness and the hardness values are reported in Table 1, where also substrate features are shown for comparison. The addition of a nitride layer results in a decrease of the surface roughness (Sa). This aspect can be very important for dies production, allowing to obtain components with a better quality surface. As for the nanoindentation results, the nanocomposite coating deposited onto nitrided H11 showed high hardness.

Table 1 Surface roughness, thickness and hardness of the coatings

| | Sa [μm] | Coating thickness [μm] | Hardness [HV] |
|-----------------|----------------------|-------------------------------------|-------------------|
| Substrate (H11) | 0,828475 \pm 0.1 | | 621 \pm 22 |
| AlSiTiN | 0,904566 \pm 0.03 | 3.1 | 2570,53 \pm 34 |
| Nit-AlSiTiN | 0,433167 \pm 0.055 | 3.1 | 3107,612 \pm 19 |

The scratch test implies elastic and plastic deformation until the coating and the substrate are separated. In general, processes of cohesive (cracking) and adhesive (flaking) failures of multilayer coatings are observed. The behaviour of AlSiTiN deposited onto nitrided H11 are shown in figure 1 while the critical loads values are given in figure 2.

Good adhesion was observed for both coatings, even if nitriding process reduces in some entity the bond between coating and substrate.

The thin film deposited over H11 substrate and over the nitrided layer showed the same type of coating failure. At the beginning, parallel cracks along the rim of the scratch channel and chipping inside of the scratch channel can be seen. The progressive propagation leads to the final complete detachment.

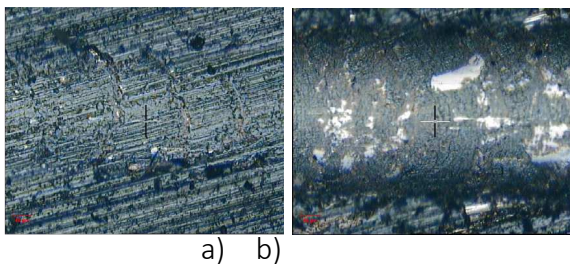


Fig. 1 AlSiTiN - nitrided H11: a) Lc1; b) Lc2

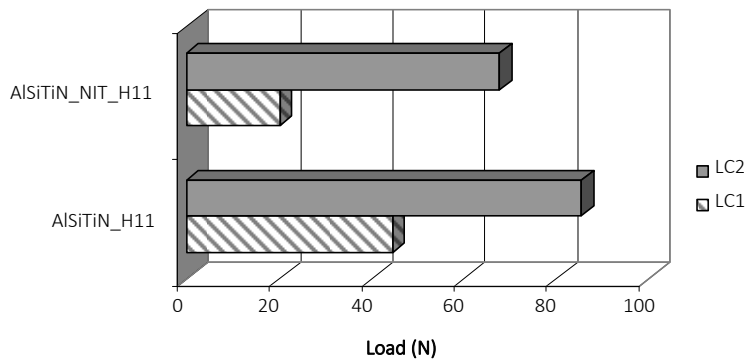


Fig. 2 Results of the scratch test in terms of critical loads.

An aluminium pin was selected for the wear test to simulate the real conditions. To evaluate wear resistance, the track after the tests was measured by optical profilometry. The depth of the wear track was obtained by measuring the height difference between a point outside the track (marked as 1 in Figure 3 a) and the bottom part of the sample trace (marked as 2 in Figure 3a). Figure 3 b shows a three-dimensional image of the AISiTiN wear track. Table 2a and 2b report the pin weight loss and the different heights at the end of the tribological test.

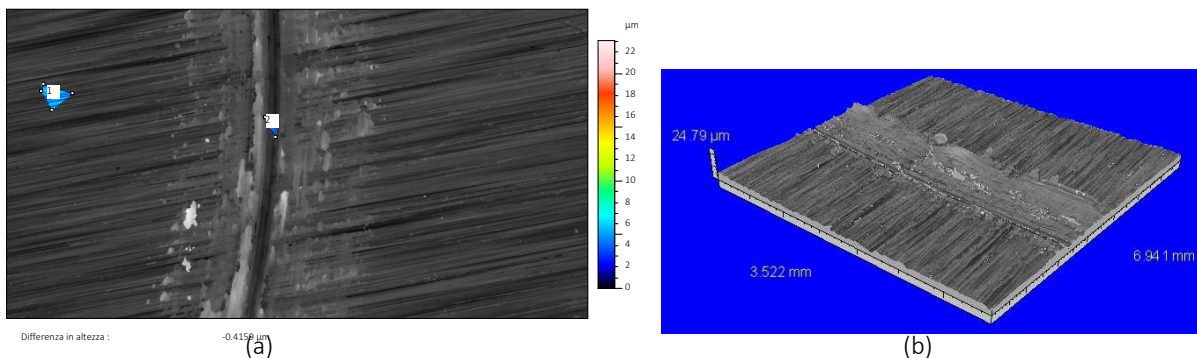


Fig. 3 a) example of methodology to measure the height variation after tribo test and b) AISiTiN wear track

Table 2 a) pin weigh loss, b) height difference

| | AISiTiN | Nit-AISiTiN |
|------------|---------|-------------|
| Weight (g) | 0.0139 | 0.0053 |

| | AISiTiN | Nit-AISiTiN |
|-------------------|--------------|--------------|
| H difference (μm) | 0,3873±0,035 | 0.3042±0,101 |

During tribological test no coating cracking or decohesion appeared within the track but, as it can be seen from images 3a and 3b, a material transfer from the Aluminium pin to the coating surface occurred during the high temperature wear test. The adhesion phenomenon was not uniform onto all the wear track, but it mainly increases at the track border and in a small part in the middle.

Comparing the height difference between the bottom part of the wear track and the external part of the wear track, (Table 2b), it can be noticed that in both case the height is widely lower the coating thickness, indicating that the substrate wasn't reached. No significant differences between the two track depths were observed, even if the ion nitrided sample showed a lower value. The wear tracks were also analyzed by SEM-EDS (figure 4 and b).

The analysis confirmed the profilometer results, indicating that the substrate wasn't reach at the end of the test. Adhesive phenomena due to the material transfer from the pin to the coating can be observed (Figure 4a), as confirmed by the elemental analysis, in which an intensity increment of the

Aluminium peak is well evident when an area affected by a soldering area is observed (Figure 4b). Oxidation phenomena are also present, as proved from the intensity enhancement of Oxygen peak in the area affected by pin adhesion.

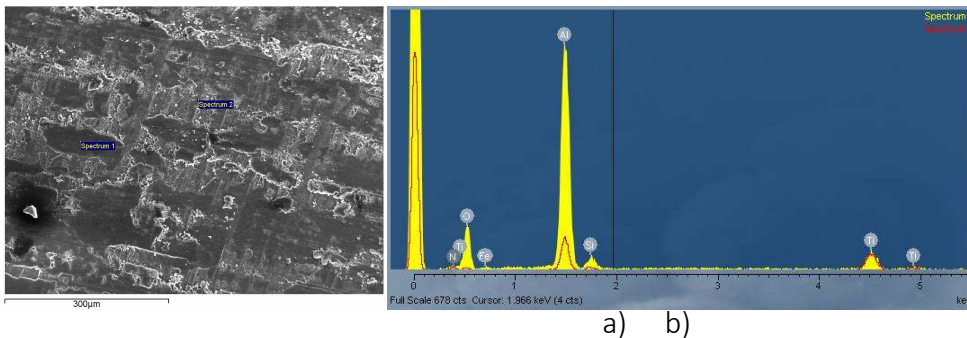


Fig. 4 SEM images and EDS analysis of the wear tracks after the ball-on-discs tests for coatings

Conclusion

In this work, the mechanical properties of a multilayer AlSiTiN coating, deposited onto H11 and nitrided H11 substrates, produced by the PVD technique, have been investigated. The results were correlated with the tribological behaviour in pin-on-disc test at 450 °C.

The nitriding step had a beneficial effect on the hardness, incrementing its value, whereas an opposite trend was observed for adhesion properties. As far as the tribological test is concerned, both samples had high wear resistance. In fact, the coating was not completely removed as a consequence of the wear tests. Nitriding caused a slight improvement in terms of wear resistance, even if the differences are not so relevant. In both cases, adhesive and oxidative phenomena caused by material transfer from the pin to the coating were observed as main wear mechanisms.

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