WEAR ON TOOL STEEL AISI M2 AND D6 COATED WITH Al_2O_3 BY MOCVD PROCESS

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***Abstract**. Present work investigates the wear resistance of tool steels coated with Al_2O_3 by MOCVD process. The wear tests by sliding and abrasion were performed in a pin-on-disc and ball-on-disc apparatus whose pin and ball substrates were tool steels made from AISI M2 and D6. The MOCVD coating processes were carried out in research laboratory apparatus at 200^oC under $N_2 + O_2$ atmosphere. The counterface discs were ABNT 1008 steel sheet used in the brazilian fridge industry. The wear resistance of the coated tool steels were evaluated trough the pin-on-disc test, using a sliding velocity 0.6 m/s, normal loads of 20N and 30N, total sliding distance of 2400 m and controlled conditions of temperature and humidity. The pin and ball material substrate were quenched and tempered, and the discs were tested as received. From the plotted graphs of lost volume versus sliding distance, it was observed that occurred a greater wear rate of AISI D6 pins without coating, this is possibly due to more severe adhesion and microcutting mechanisms. The AISI M2 and D6 pin coated with Al_2O_3 showed similar wear resistance and higher resistance than the uncoated D6 pin. However, the tested sphere of AISI M2 showed different behavior under 20N normal load. For both sphere coated with Al_2O_3 and uncoated the wear rate were similar. From microscopy observations, in order to have accurate measure of pin wear rate, it is proposed a new method to measure wear resistance of pin and ball in Pin-on-disk tests: wear can be measured by the wear track width or area left on pin tip. The graphs of track width versus sliding distance are shown and the curves for tested material and coating are compared. Pin and ball lower lost volume rate and wear track width with sliding distance is related to greater surface hardness after heat treatment and the coating process. Nitrided M2 and D6 tool steels coated with Al_2O_3 showed superior wear resistance characteristics for cold working tooling.*
***Keywords**: wear test, tool steel, stamping, MOCVD process.*

1. INTRODUCTION

In cold metal forming as sheet metal forming, piercing and blanking processes, punch and die wear out due to friction or surface and subsurface contact phenomena in sliding areas: the roughness interactions, the presence of microchips or interactions of hard particles arising from the microstructure or external contaminations with the softer surface. Besides, sheet metal shearing operations are always preceded to develop the piece contour. Generally, bulk metal forming operations can be performed by the following process: forging, rolling, direct and inverse extrusion and drawing. On the other hand, sheet metal manufacturing processes can be attained by both drawing or stretching deformations: deep drawing, stretching, redrawing or successive drawings, double or reversal drawing and stamping.

During cold metal forming operations, five basic types of tooling failure mechanisms can occur which are: wear, delamination, plastic deformation, catastrophic crack failure and cold welding. Therefore, the performance of tool steels for cold working is directly related to the mechanical properties of materials as: wear resistance, yield stress and fracture toughness. The cost involved in repairing the tooling is very high, thus, the economics implication of wear problems is obvious.

The AISI D6 and HSS M2 tool steels are commonly used in punch and die for cold forming operations, therefore, they were utilized in the present research work(1). In addition, advanced surface engineering methods have been developed to improve the surface mechanical properties while maintaining good toughness in the bulk(2). These new emerging techniques can be surface coating as the PVD and CVD processes or surface modifications as nitriding. More recently, the MOCVD process (metal organic chemical vapor deposition) has been applied to coat several metal substrates(3).

In summary, a comparative study of wear resistance of AISI M2 and D6 tool steels under normal heat treatment and coated with Al_2O_3 by MOCVD process against discs of ABNT 1008 steel as counterface has been carried out. ABNT 1008 steel sheets are used in the fridge industries and M2 and D6 tool steels in cold forming. Wear performance and wear mechanisms were experimentally investigated, using the Pin-on-disk methodology and the scanning electronic microscopy micrographs.

2. EXPERIMENTAL PROCEDURE AND MATERIALS

Wear resistance performance of materials are commonly obtained from testing carried out in pin-on-disk equipment, according to ASTM G 99-95 standard procedure. It gives a laboratory standard method to carry out sliding and abrasion wear tests. Each experimental test uses a set of selected constant operation conditions: total sliding distance, normal load on the pin and the sliding velocity(4,5). Table 1 shows the utilized parameters during the tests in the present investigations.

2.1 Test Specimens

2.1.1 Pin and Ball

The pin test specimens were made from AISI D6 and HSS M2 tool steels heat treated, HSS M2 steel heat treated and nitrided. The fabrication of the pins were performed by conventional methods as machining and milling to obtain the appropriate geometry with a spherical tip which radius was approximately 10 mm. See Figure1 and Figure 3 for geometry. After machining, the pins were heat treated to increase its hardness, according to tooling requirements. Some pins were coated with Al_2O_3 by the MOCVD process.

The sphere substrate specimens were commercial steel balls made from HSS M2 steel with 10mm in diameter. They were tested as received and with the Al_2O_3 coating. Table 2 shows the hardness of the pin and balls substrates, the Al_2O_3 film hardness and process temperature.

The Al_2O_3 coating by the MOCVD process on the steel substrates were carried out in a research laboratory apparatus at temperature 200^oC under $N_2 + O_2$ atmosphere(3) .

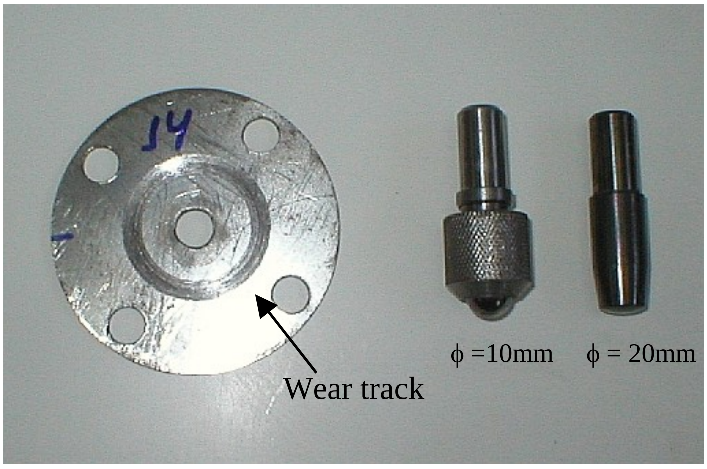


Figure 1. Disk, ball and pin used in the pin-on-disk wear testing.

Table 1. Parameters utilized in the pin-on-disk testing.

Sliding velocity (m/s)	Load (Kgf)	Sliding distance (m)	Track radius (mm)
0.6	1.930 and 2.953	2400	14.3

Table 2. Nominal characteristics of balls and pins substrates coated with Al_2O_3.

Material	Microhardness [HV 0.05]	Film thickness (micron)	Process temperature (^oC)	Friction coef. against steel
Substrate M2	770	----	----	0.4
Substrate M2 nitrided	1300	----	500°	0.4
Substrate D6	800	----	----	0.4
Film Al_2O_3	2500	0.500	≤ 200°	0.2

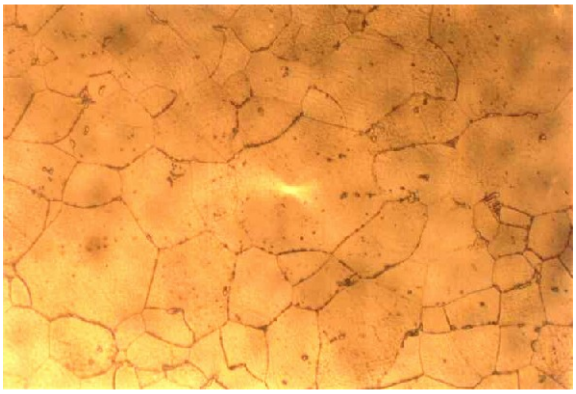


Figure 2. Surface micrograph of ABNT 1008 steel. 200x.

Table 3. Hardness and geometric features of disk counterface.

Material	Vickers Hardness kgf/mm^2	Thickness mm	Roughness Ra
ABNT 1008 Steel	110	0.5	200 μin

By using aluminum dimethylisopropoxide as the precursor, varying the reaction conditions (such as temperature and total pressure) and, most important, by using water vapour as the reacting gas, it was obtained high density, transparent aluminum oxide films with extremely smooth surface texture, at growth temperatures as low as 180°C, and a growth rate of up to about 150 nm min^{-1}. A reaction mechanism has been proposed for the precursor decomposition, stressing the important role of water vapour in the decomposition process.

The substrates were cleaned prior to the introduction into the reactor: they were immersed in soaped water, washed with distilled water, rinsed in isopropylic alcohol and dried in air.

2.1.2. Disk

The counterface or disks, see Figure1 and Figure 4, were obtained by shearing ABNT 1008 mild steel sheets as received and they have 0.5 mm in thickness by diameter 62 mm, as used in industry. The disk was cut in the appropriate dimensions for testing and contained 5 holes with diameter 8 mm that were machined to fix the disk in the base. In table 3 the hardness and geometric characteristics of the steel disks can be observed. Figure 2 shows the microstructure of steel disk surface. Regular equiaxial grains distribution and size are observed.

2.2. Procedure for Pin-on-disk Testing

Initially, the specimens followed a rigorous preparation procedure in order to eliminate any trace of dust, debris or oxidation, using a flux of compressed air, ethylic alcohol and a clean cloth. After, pin and disk were weighted in an analytical balance, with 0.0001 g of accuracy, for determining its initial mass before the tests. Then, pin and ball were fixed in exactly the same position as initially marked.

The pin-on-disk apparatus parameters as the track radius, disk rotation and the revolutions counter were adjusted to the selected conditions: see Table 1 and Figure 3 for equipment sketch. The pin-on-disk apparatus was equipped with a large glass campanula that covered the specimens. Temperature and humidity inside the campanula were kept at approximately 25°C and 55 to 60% of relative humidity. Two types of normal loads on pin or ball were selected for each test: 20 N and 30 N. The counter was set to stop the test at every 200 m of pin sliding distance in order to allow measuring the intermediate mass lost for pin and disk. Each test was considered completed for 2400 m sliding distance. A full specimens cleaning, using a flux of compressed air and a cloth with ethylic alcohol 99,5% always preceded these measurements. Before weighting, the specimens were dried out in a furnace at 80°C for 10 min to avoid any solvent in the specimen and to evaluate the real mass lost from the pin, ball and disk.

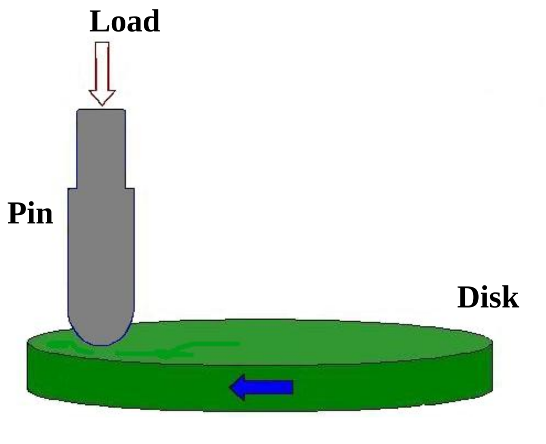
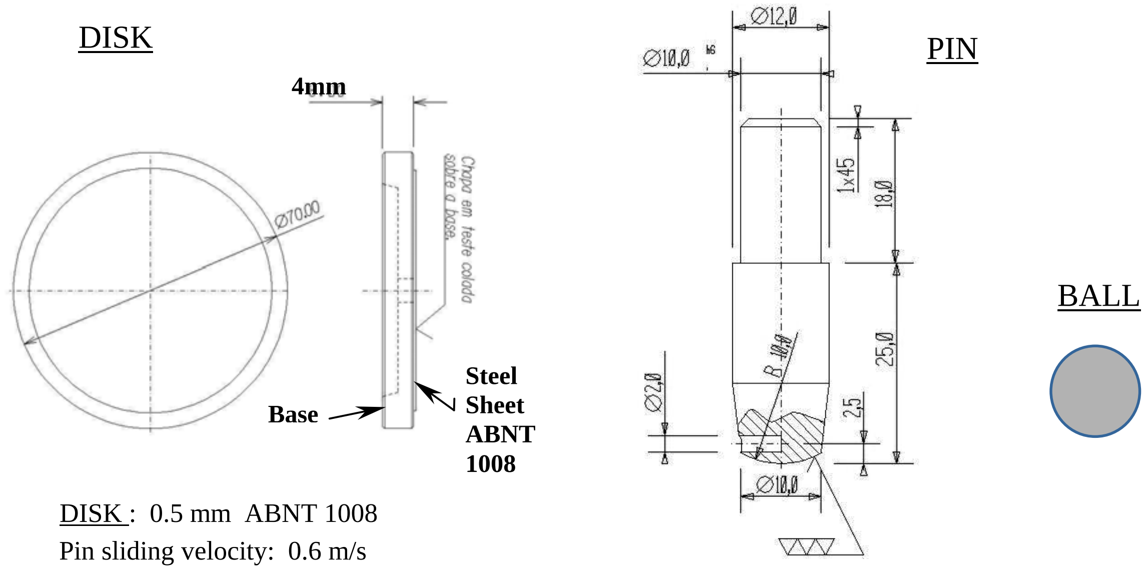


Figure 3. Skecth of Pin-on-Disk apparatus.



DISK : 0.5 mm ABNT 1008
Pin sliding velocity: 0.6 m/s
Balance : accuracy 0.0001 g
Normal atmosphere conditions :
25°C and 55 to 60% relative humidity.

PIN: AISI M2 and D6 Tool steel; ϕ=20mm
BALL: M2 Tool steel ; ϕ = 10 mm
Normal loads: 20 N and 30 N

Figure 4. Disk, Pin and Ball geometry used as specimens in the Pin-on-disk apparatus.

3. RESULTS AND DISCUSSIONS

The experimental results of Pin-on-disk and Ball-on-disk tests for pin and ball lost volume versus sliding distance are presented in Figure 5 and Figure 6. Lost volume was calculated by the division of measured lost mass by the substrate density. Results are for lost volume for each specimen sliding against ABNT 1008 steel disk for total distance 2400 m and normal loads 30 N and 20 N respectively. The wear rate (Q = lost volume/sliding distance) is different for each substrate and coating, according the Archad equation[2,6] for abrasive and sliding wear,

$$Q = K \frac{P}{H} \quad (1)$$

where K is the wear coefficient (1/K is the wear resistance), P is the normal load, H is the hardness of the softer material (disk).

The experimental curves shown below are roughly linear, but some materials present a trend to decrease the wear rate at the final stage. This is possibly due to the decrease in the nominal pressure under the pin tip or ball, as the contact area increases and the normal load remains constant. Expected contact pressure on sphere are superior to the pin due to its lower radius of tip contact: tip radius is 5 mm for sphere and 10 mm for pin. This caused the greater were rate of the balls compared to pins.

It is observed that the balls wear rate almost double when the load increased from 20 N to 30 N. The coated pins have shown superior wear resistance, i.e. lower wear rate, than the uncoated pins. However, the wear rate of the coated M2 steel ball is quite similar to the uncoated ball for load of 30N.

In Figure 6, pin and ball lost volume with the sliding distance is presented for normal load of 20 N. The curves are irregular, indicating a non-linear wear rate. Apparently, from the observations of Figure 5 and Figure 6, the uncoated balls have higher wear resistance. This is certainly due to the adhesion mechanisms of disk material on pin and ball as seen in Figure 10 and Figure 11.

As expected, the Al_2O_3 protective coating has substantially improved the wear resistance of AISI D6 steel pin as shown in Figure 5. Coated pins, D6 and Nitrided M2, have quite similar wear resistance.

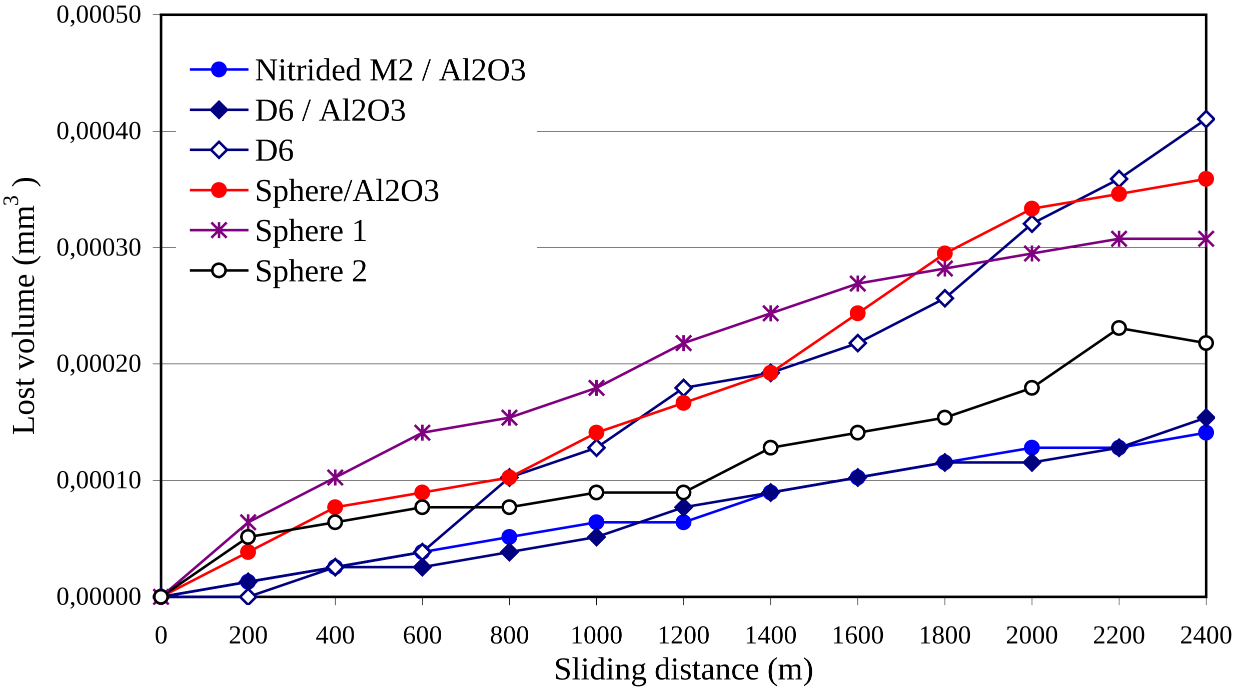


Figure 5. Pin-on-disk experimental results. Pin and Sphere lost volume versus sliding distance. Normal load 30 N.

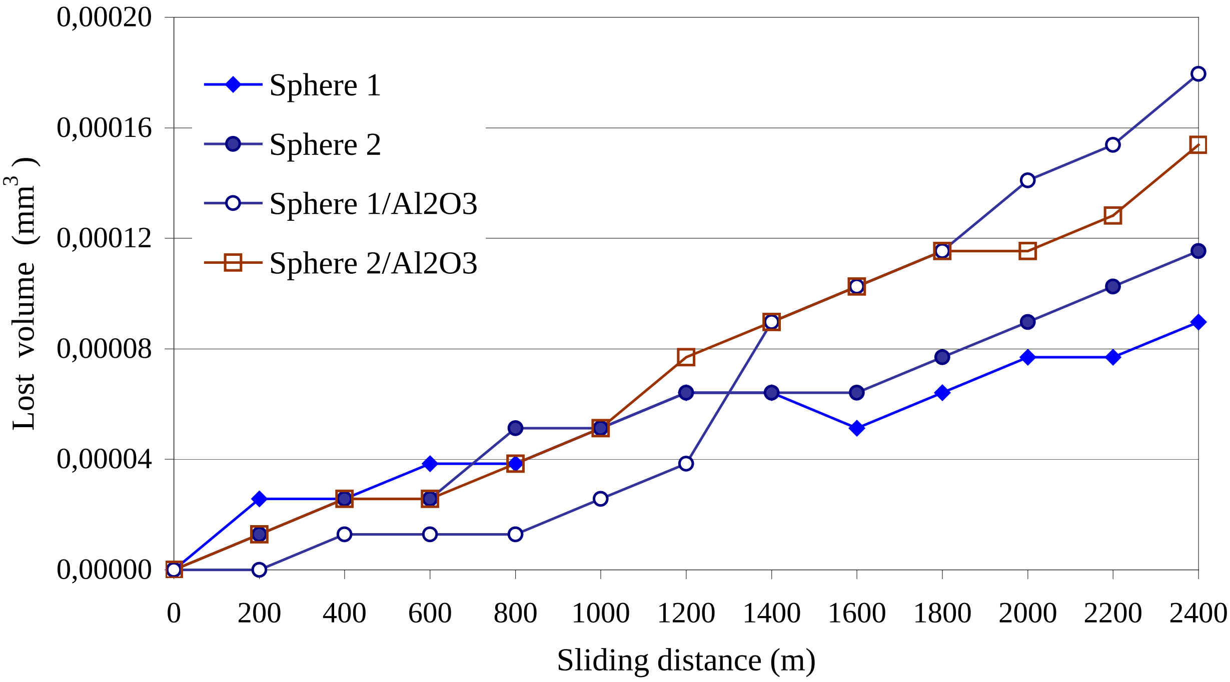


Figure 6. Pin-on-disk experimental results. Pin and Sphere lost volume versus sliding distance. Normal load 20 N.

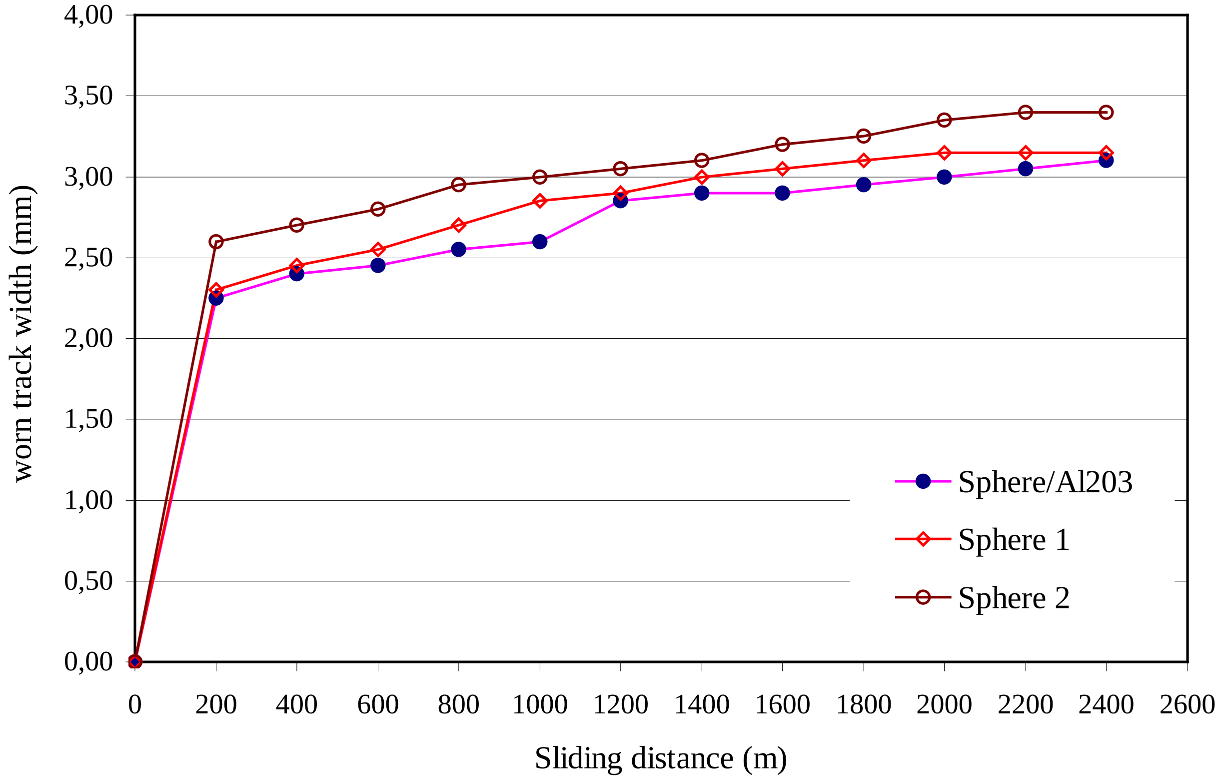


Figure 7. Evolution of sphere (AISI M2) worn track width w versus sliding distance. Normal load 30 N.

In order to investigate a better measurement method for balls and pins wear, the worn area width of pin and ball were measured as shown in Figure 9. The wear contact area of pin and ball had an elliptical shape and can be accurately measured, using a CCD camera connected to a monitor.

A simpler method is to determine the major diameter length or track width w as seen in Figure 9. The curves in Figure 7 are better defined and more linear than the curves for lost volume. By this method, as expected, the ball with Al_2O_3 protective coating had lower wear rate than the uncoated balls as observed in Figure 7. However, the wear resistance of Al_2O_3 coating is somehow weaker than expected for the present test. This is possibly due to the film thickness (0.5 micron) and its low adhesion resistance to the steel substrate. The Al_2O_3 film is broken at the initial sliding stages of the wear test as seen in Figure 11. The sequence of SEM micrographs taken at 50 m and 100 m of sliding distance shows the wear mechanisms of the Al_2O_3 film as: microcraking, delamination and material adhesion. Although, abrasion mechanisms of substrate are observed at 2400 m as noted in Figure 9.

In Figure 7, a tendency of the wear track width to remain constant, thus the wear rate to fall to zero, as the sliding distance approaches the 2400 m it is noted. This is due to the decrease in the nominal and real contact pressure on the ball as the groove or track formed in the disk increase.

In Figure 8, the Al_2O_3 film surface features can be analyzed. The occurrence of particles or blisters under the film is seen in Figure 8a. Scratches and pores in the substrate are observed in Figure 8b. Thus, the film has not been enough to fully cover the substrate roughness. These substrate defects at surface have possibly influenced the Al_2O_3 film wear resistance.

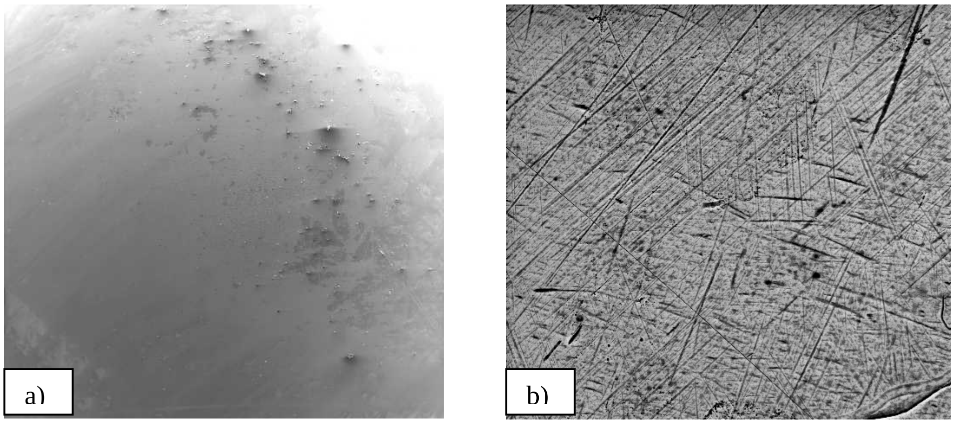
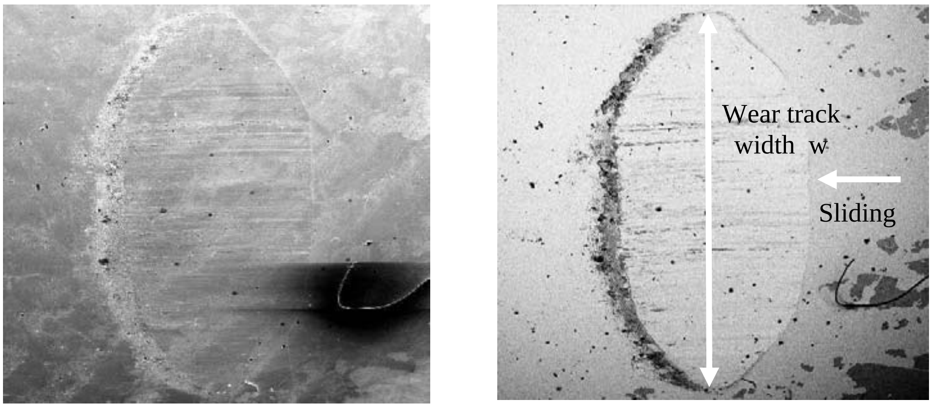
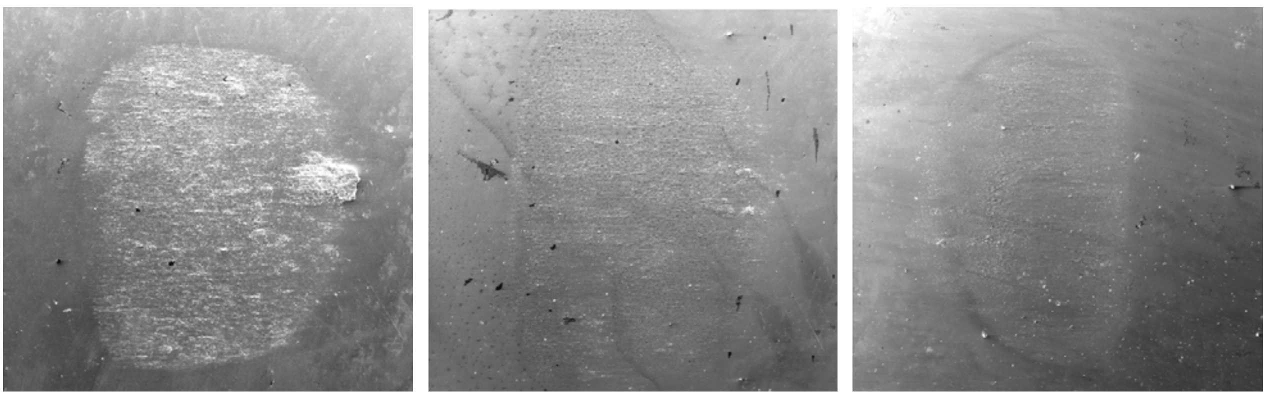


Figure 8. The Al_2O_3 coating over sphere before wear test: a) 50x and b) 1000x. Roughness features of the sphere substrate surface remains on the coating surface. Al_2O_3 film did not filled the micro-groves.



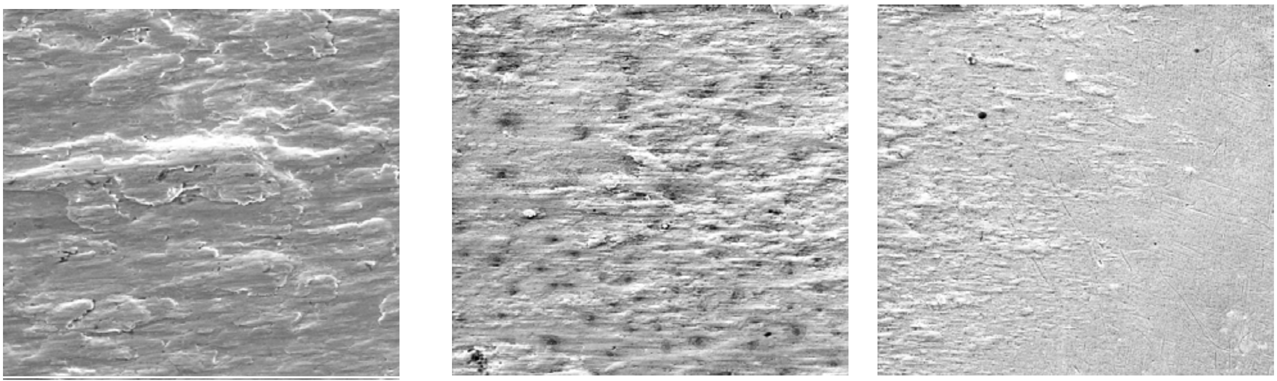
a) Sphere micrograph SE - 30x   b) Sphere micrograph BSE - 30x

Figure 9. SEM micrographs of the Al_2O_3 coating over sphere after wear test: a) SE, 30x and b) BSE, 30x. Wear track geometry of the worn coating can be measured. Load 20 N, sliding distance 2400m.



a) Sliding distance 50m. 50x.   b) Sliding distance 100m. 50x   c) Sliding distance 150m. 40x.

Figure 10. SEM micrographs of the Al_2O_3 coating on sphere at intial wear test for load 20 N. Wear track geometry of the worn sphere tip can be measured. Sliding distance at 50m, 100m and 150m.



a) Delamination, micro-cracking b) Delamination, adhesion c) Adhesion

Figure 11. Evolution of wear behaviour of Al_2O_3 coating. SEM micrographs of wear mechanisms with sliding distance for normal load of 30 N. a)50m -1000x ; b)100m -500x ; c)100m -500x.

4. CONCLUDING REMARKS

From the experimental observations and results in the present work, some relevant conclusions can be drawn about wear on Al_2O_3 coating , AISI D6 and HSS M2 tool steels.

Pin-on-disk tests and Scanning Electron Micrographs (SEM) observations provided important insights into the wear behaviour of coating Al_2O_3 deposited by MOCVD process on tool steels.

Initial wear mechanisms of coating have been observed on the sphere contact surface against the disk after the initial 150 m sliding distance and for normal load 20 N. The main observed wear mechanisms were: micro-cracking, delamination and adhesion.

The curves of pin and sphere lost volume versus sliding distance are reasonable linear for the load 30 N and irregular for the load 20 N. Thus, the tests with load 30 N are preferable for wear resistance investigations of tool steels as the wear rate remain constant along the sliding distance.

For the same load and material, the wear rate depended on the pin tip radius.

The Al_2O_3 thin film has not increased the wear resistance of AISI M2 tool steels (sphere). However, increased the wear resistance of AISI D6 (Pin).

From microscopy observations, it is proposed a new method to measure wear resistance of the pin in Pin-on-disk tests: wear can be measured by the wear track width or area left on pin tip. In present work, the wear track width was almost linear with the sliding distance.

In general, the quality of the coatings deposited by MOCVD process depended upon the adhesion strength to the substrate, roughness of substrate, porosity and film thickness. Residual hard particles, substrate roughness and film thickness influenced the coating tribological performance. Wear morphology changed with the sliding distance.

The wear performance of AISI M2 coated with Al_2O_3 film has been improved when measured by the wear track width w versus sliding distance instead of calculating the lost volume versus sliding distance. Nitrided M2 tool steel and coated with Al_2O_3 has superior wear resistance.

5. ACKNOWLEDGEMENTS

The authors would like to gratefully thank the financial support given by CNPq of Brazil by a research scholarship, as well as CAPES, SOCIESC and University of Santa Catarina State – UDESC.

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DESGASTE EM AÇOS FERRAMENTA AISI M2 E D6 REVESTIDOS COM Al_2O_3 POR PROCESSO MOCVD

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***Resumo**: O presente trabalho investiga a resistência ao desgaste de aços ferramentas revestidos com Al_2O_3 pelo processo MOCVD. Os ensaios de desgastes por deslizamento e abrasão foram realizados num equipamento tipo pino-sobre-disco cujo material do substrato do pino foram de aços ferramenta AISI M2 e D6. O processo MOCVD de revestimento foi feito num equipamento de laboratório na temperatura de 200^oC e atmosfera de N_2 + O_2 . Os discos de contraface foram chapas de aço ABNT 1008 usadas na indústria brasileira de refrigeradores. A resistência ao desgaste dos aços revestidos foi avaliada através do teste de pino-sobre-disco, utilizando uma velocidade de deslizamento de 0,6 m/s, carga normal de 20N e 30N, distância total de deslizamento de 2400 m e condições controladas de temperatura e umidade. Os materiais do pino e da esfera foram temperados e revenidos, e os discos ensaiados como recebido. Dos gráficos traçados de volume perdido versus distância de deslizamento, observou-se que ocorreu uma maior taxa de desgaste dos pinos de AISI D6 sem revestimento, isto é possivelmente devido a mecanismos mais severos de aderência e micro-usinagem. Os pinos AISI M2 e D6 revestidos com Al_2O_3 apresentaram resistência ao desgaste semelhante, mas superior a do pino D6 sem revestimento. Entretanto, as esferas ensaiadas de AISI M2 mostraram um comportamento diferente sob carga de 20N. Para ambas esferas revestidas com Al_2O_3 e não revestida, a taxa de desgaste foram semelhantes. Das observações no microscópio, a fim de se ter medidas mais precisas da taxa de desgaste dos pinos e esferas, propõe-se um novo método de se medir a resistência ao desgaste nos ensaios de pino-sobre-disco: o desgaste pode ser medido através da largura ou área visível de desgaste deixada na ponta do pino ou esfera. As curvas da largura do desgaste versus a distância de deslizamento são mostradas para os materiais e revestimento comparadas. As menores taxas de volume perdido e largura do desgaste nos pinos ao longo da distancia de deslizamento está relacionada com dureza superficial maior após o tratamento térmico e o revestimento. Os aços ferramenta M2 nitretado e D6 revestidos com Al_2O_3 apresentaram resistência ao desgaste superior para aplicação em ferramentas de conformação à frio.*
***Palavras chave**: ensaio de desgate, aço ferramenta, revestimento de alumina, MOCVD.*