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Laser surface texturing of PVD coatings applied to sheet forming dies for stainless steel

M. Calandri, D. Ugues, M. Lorusso, A. G. Demir, N. F. Lecis, B. Previtali

Fiber laser surface texturing was applied on Cr nitride based and other PVD coatings. The field of interest of this study is the cold forming of stainless steel sheets. The laser surface textured coatings were initially investigated in terms of structural and tribological properties in order to select the coating system which achieved the best benefits from laser machining. This set up process was performed at laboratory level traditional pin on disk tests using stainless steel as counterpart. The test conditions were set up to generate severe compressive loads and low sliding velocity with boundary lubrication conditions, thus favouring adhesive wear and stainless steel transfer onto the textured coated surface. Results in terms of wear coefficient of friction, gain/losses, material transfer and structural properties are given so as to identify the optimal coating grade to be subject to laser surface texturing.

KEYWORDS: LASER SURFACE TEXTURING - HARD PVD COATINGS - COLD FORMING DIES - STAINLESS STEEL SHEET FORMING - POOR LUBRICATION CONDITIONS

INTRODUCTION

As environmental restrictions on the use of lubricants become tighter and costs associated with their disposal increase, there is a growing demand for low friction materials that allow mating surfaces to slide one against each another with reduced friction and wear [1]. This is particularly critical in load bearing components such as sliders, sliding bearings and cold forming tools which are prone to severe adhesive wear as they are commonly subjected to heavy normal loads and low-to-medium sliding speed. Surface texturing is one of the viable methods for enhancing tribological properties of mechanical components. Amongst different processes used, laser surface texturing (LST) is an established method, which is utilized to generate micro pockets, commonly in form of micro dimples, on the surface of the mechanical component that mainly act as lubricant reservoirs during the sliding contact [2]. In addition to providing the necessary lubricant release during contact they can also entrap the worn particle and debris to reduce wear provided by third body effect. The overall effect is the reduction of the friction and wear, resulting in lower energy consumption and increased lifetime of the component. Apart from tribological applications, wetting [3] and optical [4] properties of the textured surfaces, nanostructure formation inside dimples formed by ablation [5] are other points of interest in laser surface texturing research. Compared to other methods to texture surfaces, laser process provides high flexibility of the obtainable geometries with elevated precision, and excellent control of the shape and size. Furthermore, it allows short preliminary preparation and post processing, as it does not require adapted dies or tools and it is environmental friendly.

Laser texturing has been applied to various tribological systems based on metallic materials, including the ring/liner assembly of engines, mechanical seals and bearings. LST has been shown to reduce friction coefficient by 30% in piston ring applications [6,7]. A similar positive effect of laser surface texturing on frictional performance of face seal was also demonstrated [8]. Under boundary lubrication regime LST was observed to expand the range of the hydrodynamic lubrication regime in terms of load and sliding speed for both high- and low viscosity oil lubricants [9]. Promising results have been also reported in the field of WC/C turning cutting tools recently [10]. Besides the traditional applications on metallic materials, the use of LST was also expanded to ceramic surfaces where tribological behaviour was improved compared to un-textured polished surfaces [11]. From the point of view of the used laser source several types differing in wavelength and pulse width

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have been used for laser surface. In particular, CO_2, Nd:YAG, excimer lasers, operating in fundamental wavelengths or other harmonics, with *ms* to *ns* pulse widths have been employed [12-17]. More recently, the research in the field has been guided towards new trends of reducing the dimension of textured features, application of solid lubricant inserted into laser formed micro-reservoirs and moreover to the use of ultra fast lasers operating with *ps* and *fs* pulses. It was reported that the wear life of burnished MoS_2 film in laser textured nickel-based composite surfaces was found to be significantly higher than that for the same film on plain surfaces [18]. Laser surface textured TiCN coatings with 10-20 µm dimples were also reported to be filled with solid lubricants using burnishing and sputtering, being such practise capable to highly improve the wear properties compared to the monolithic TiCN [19]. In such studies the tribological properties of LST coatings were studied in point contact configuration. It is known that due to their relatively shorter pulses, *ps* and *fs* lasers reduce thermal damage compared to nanosecond lasers [20]. Furthermore, *fs* laser ablation has been studied for the crater formation and hydrostatic properties of single craters [21].

Despite of the extensive research, laser surface texturing has not been widely applied in industry. As a process which consists of generating some thousands of micro craters on relatively large surfaces, LST requires a stable and robust production tool, giving high productivity, flexibility, reduced capital and maintenance costs so as to meet the economy of scale required by industrial use. Pulsed fiber laser systems operating with nanosecond pulses are a promising new option for LST, as they can provide high productivity along with sufficient quality. Fiber lasers are characterized by high brilliance and high focusability, resulting in beams of high quality and very small diameter; high pulse energies and repetition rates allow higher productivity. Due to their simplicity, robustness and reduced cost, they are appealing for industrial applications. Application of pulsed fiber lasers to LST is a very recent subject, it has been rarely reported on metallic materials [22], and the tribological performance of generated surfaces is so far inexistent in the literature.

In this paper a study on potentials of applying laser surface texturing on hard coatings was given. The final intended application for this material design is the cold forming of stainless steels sheets. In this case heavy loads, low sliding speeds and the chemistry of the workpiece material favour severe adhesive wear. Lubricant oil is typically used, but under heavy load conditions adhesion can occur anyway especially in very complex die profiles. Additionally the needs to reduce the use of lubricants is pushing to work in poorly lubricated working conditions, thus favouring the microscopic contacts between surfaces.

MATERIALS AND METHODS

Substrate material used for all the characterization was cold work tool steel n. 1.2379 (AISI D2). Vacuum heat treatment was applied: austenitizing at 1070°C, 30 minutes; quenching in nitrogen flux at 9 bar; first annealing at 520°C, 90 minutes; second annealing at 470°C, 90 minutes. Surface hardness in quenched and tempered state was 746 HV.

This material was used to fabricate wear test samples in the shape of disks (diam. 30 mm, height 10 mm) for standard pin on disk test. The same type of samples was used also to investigate coatings structural features and to perform Laser Surface Texturing trials. Before coatings deposition was applied grinding and polishing of steel samples was performed so as to achieve a fine finishing level (Ra=0.02 ± 0.01) on substrate surfaces.

Four different coating grades were then applied through Cathodic Arc Evaporation Physical Vapour Deposition (CAE-PVD) process. Nominal features of these coatings are reported in Table 1.

Tab. 1 - Coating grades and key features

Coating grade	Nominal Microhardness [HV 0.05]	Coefficient of Friction (dry)
AlCroNa (AlCrN) - BAP	3,200	0.35
Lumena (TiAlN) - BL	3,400	0.30-0.35
Futura (TiAlN) - BFN	3,300	0.30-0.35
Futura Advanced (TiAlN) - BFN Ad	3,300	0.25

Coatings morphology was evaluated on some samples through Scanning Electron Microscopy (SEM) in top and transversal view, in order to assess for each grade the level of defects present in different coatings, the rough chemical analysis (through Electron Dispersive Spectroscopy) and the thickness. Nanohardness and reduced Young modulus were measured using a Hysitron Nanoindenter 950T. For nanohardness study 16 indentations were performed on each coating grade setting increasing indentation load from 1000 µN to 10000 µN in steps of 600 µN. The maximum level of indentation load was maintained for 2 seconds. The indentations were made at 5 µm distance one from each other and for every indentation a load application speed of 200 µN/s was adopted.

Further to this structural verification a set of coated samples was subjected to laser surface texturing (LST). An IPG Photonics 1/100/50/50 active fibre laser machine and a Century Sunny TSH 8310 scanner with 100 mm focal lens were used. The laser system was set up to the following values: 1) pulse duration: 250 ns (100 ns FWHM); 2) Maximum energy: 1 mJ; 3) Beam diameter at focal point 39 µm. LST processing was carried out according to the parameters reported in Table 2.

Tab. 2 - Laser surface texturing parameters belonging to the different surface coatings.

Coating	PI%	PRR	Tmod [µs]	hf [mm]	Thickness [µm] *	Dimple Depth [µm]
BAP	100	50	65	1.1	3.0	2.37±0.05
BFN Ad	100	50	65	0.7	9.0	4.67±0.15
BFN	100	50	65	1.3	3.5	1.56±0.11
BL	100	50	65	0.5	10.0	7.29±0.14

*Estimated via machining trials.

The laser systems used in this work operated in the nanosecond pulse regime. During the LST process it was very important to avoid the complete penetration of the coating and the consequent mixing between coating and substrate. This limitation of dimple's deepness resulted also in a limitation to the dimple's diameter because an adequate aspect ratio value must be respected.
Samples simply coated with the different coating grades and further laser surface textured was subjected to wear test. Lubricated pin on disk tests were applied using an AISI 304 stainless steel pin with an hemispherical end cup of 3 mm radius and a central blind hole on the opposite side used as thermocouple's location so as to monitor the pin temperature along the wear test. Before to perform the tests the samples were covered with oil (Multidraw KTL N 16 LM) through a brush. Sliding speed was 0.5 m/s and normal contact load 20 N. The total sliding distance was 2000 m, but the coefficient of friction was evaluated only during the first 1000 m. This method was adopted since in the major part of the tests the pin wear was so high that the contact condition between pin and disc changes. This is of course due to the fact that the hardness of coated surfaces and counterpart pins were very different, thus resulting in rapid wear of the soft pin. Nevertheless this configuration was selected to better resemble the final application service condition: cold forming of stainless steel sheets, where plastic deformation of the metallic sheet is locally imposed, thus generating potential adhesion onto the die surface. To tackle this aspect, apart from friction coefficient and weight losses, the tendency of counterpart material to transfer on coated and LST coated surface was measured through SEM investigations and image analysis treatments.

RESULTS AND DISCUSSION

The morphology of the four type of coatings used in this study is shown in Figures 1 as top view and transverse section mode. Every coating has local defects, such as porosity and droplets, that are typical features of the CAE-PVD process.
The analysis in terms of number and dimension of pin-holes defects was realized through the use of an image analysis software, Gwyddion 2.30. In Figure 2 the number of defects recorded per each coating grade is reported.
Defects detection was performed through SEM images (2,500x) captured in 10 random locations on top view coated surfaces. Every coating has a typical columnar structure and different thickness. Thickness' values of the four coating grade are reported in table 3.

Tab. 3 - Thickness' values of each coating grade (estimated by transverse section SEM images)

BAP [µm]	BL [µm]	BFN [µm]	BFN Ad [µm]
2	13	4	8

The chemical compositions of the four types of coating as measured by EDS analysis are reported in Table 4. The average values of the roughness parameters Ra and Rz of the steel substrate and of the four types of coatings are reported in Table 5 with their relative standard deviations.

Tab. 4 - Chemical composition of each kind of coating gained by EDS (W = weight, A = atomic)

Sample	Nitrogen (N)		Aluminum (Al)		Chromium (Cr)		Titanium (Ti)	
	W (%)	A (%)	W (%)	A (%)	W (%)	A (%)	W (%)	A (%)
BAP	23.27	45.56	23.68	24.06	53.01	30.35	-	-
BL	25.96	47.48	35.18	33.41	-	-	37.90	18.68
BFN	22.50	44.56	23.56	24.22	-	-	53.69	31.09
BFN Ad	21.68	43.27	24.41	25.28	-	-	53.59	31.28

Tab. 5 - Average values of Ra and Rz and their relative standard deviations recorded on substrate and on each coating type.

Sample	R_a [µm]	Standard deviation [µm]	R_z [µm]	Standard deviation [µm]
Substrate	0.02	0.01	0.18	0.06
BL	0.15	0.02	1.53	0.32
BAP	0.05	0.01	0.46	0.05
BFN	0.07	0.02	0.52	0.13
BFN Ad	0.15	0.02	1.34	0.26

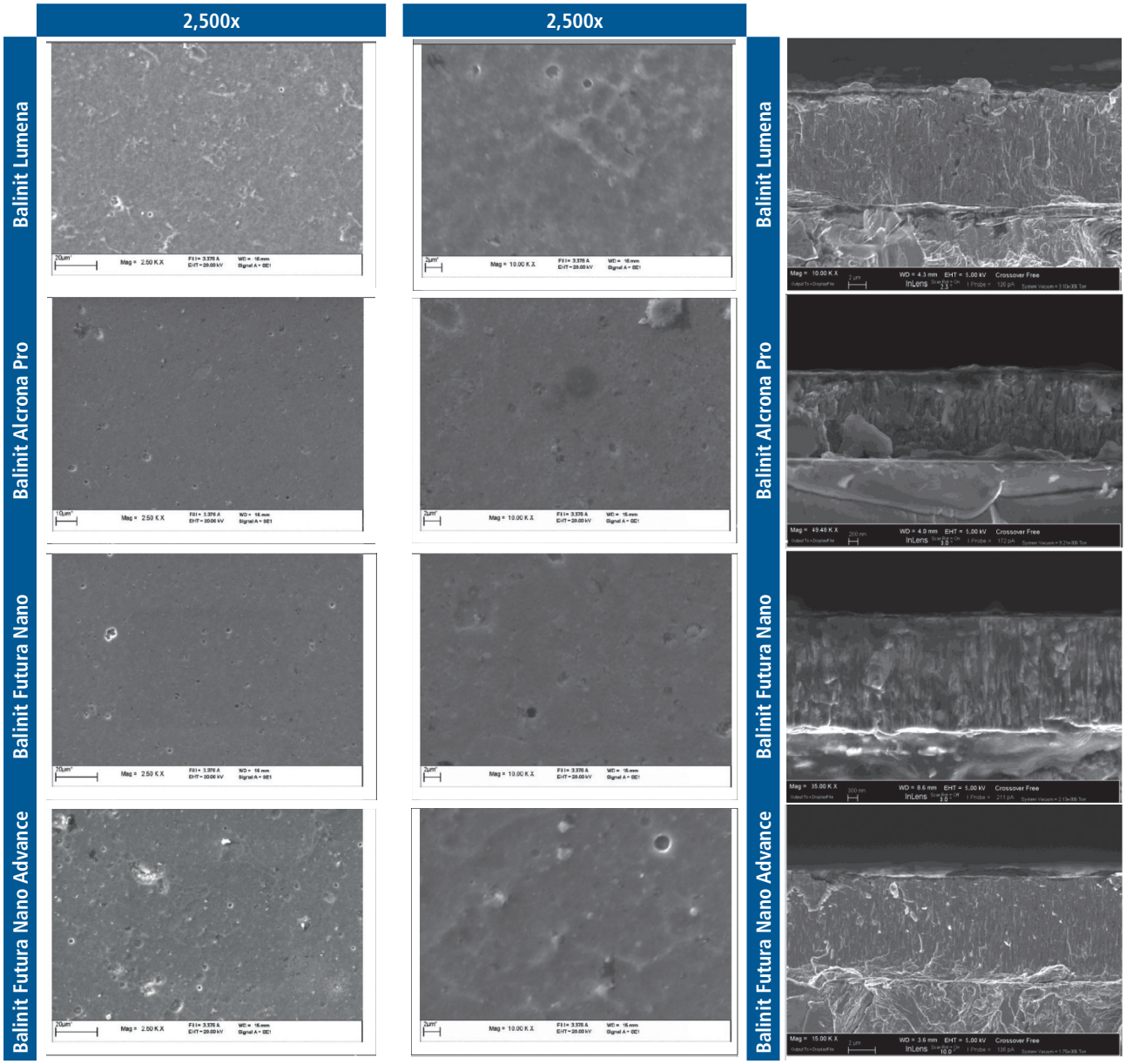

Fig. 1 - SEM images in Top View configuration of each coating at magnification of 2,500X (marker: 20 µm) and of 10,000X (marker: 2 µm)

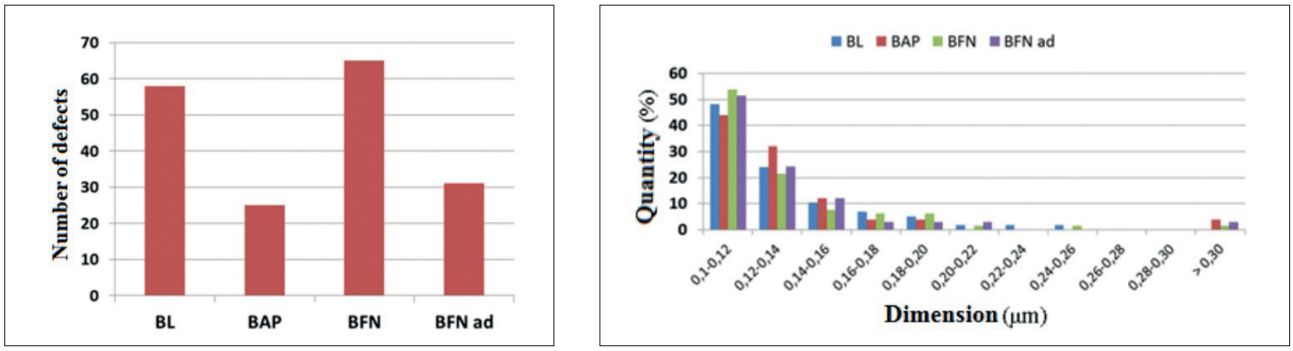

Fig. 2 - Total number of defects for each coating grade and the relative size distributions of such defects

The reduced Young's modulus and the Nanohardness of every coatings were obtained through Nanoindentation tests, results being reported in Table 6. It is worthwhile to note that standard deviations recorded on coated surface are typically much higher than those recorded on uncoated samples. Such fluctuations were attributed to coating's in-homogeneities in terms of surface defects.

Tab. 6 - Average values of the reduced Young Modulus and of Nanohardness and related standard deviations for substrate and for each kind of coating

Sample	Reduced Young modulus [GPa]	Standard deviation [GPa]	Nanohardness [GPa]	Standard deviation [GPa]
Substrate	216	13	8.43	0.67
BAP	356	61	30.03	8.38
BFN	328	36	27.49	6.09
BFN Ad	305	36	24.99	5.66
BL	333	56	26.25	7.67

In the following pictures the results provided by Laser Surface Textured samples are reported. Figure 3 reports the results recorded on each coating grade. The samples identifying codes refer to the type of coating and the conditions of laser texturing applied. Apart from BAP (i.e. Balinit Alcrona) and, to some extent, from BFN (i.e. Balinit Futura), the coatings did not react positively to laser surface texturing, at least in terms of Coefficient Of Friction (COF). Actually in the case of BAP and BF it was actually possible to measure a reliable average value of COF. For BAP the COF was lower than 0.2, whereas for BFN was lower than 0.3. These values are really similar to those recorded in the same wear test on the steel substrate and on the same plain coating grades. For the other two coatings the COF fluctuation was so high in some of the analysis that COF was meaningless. This will be attributed to the excessive pin wear recorded in such tests.

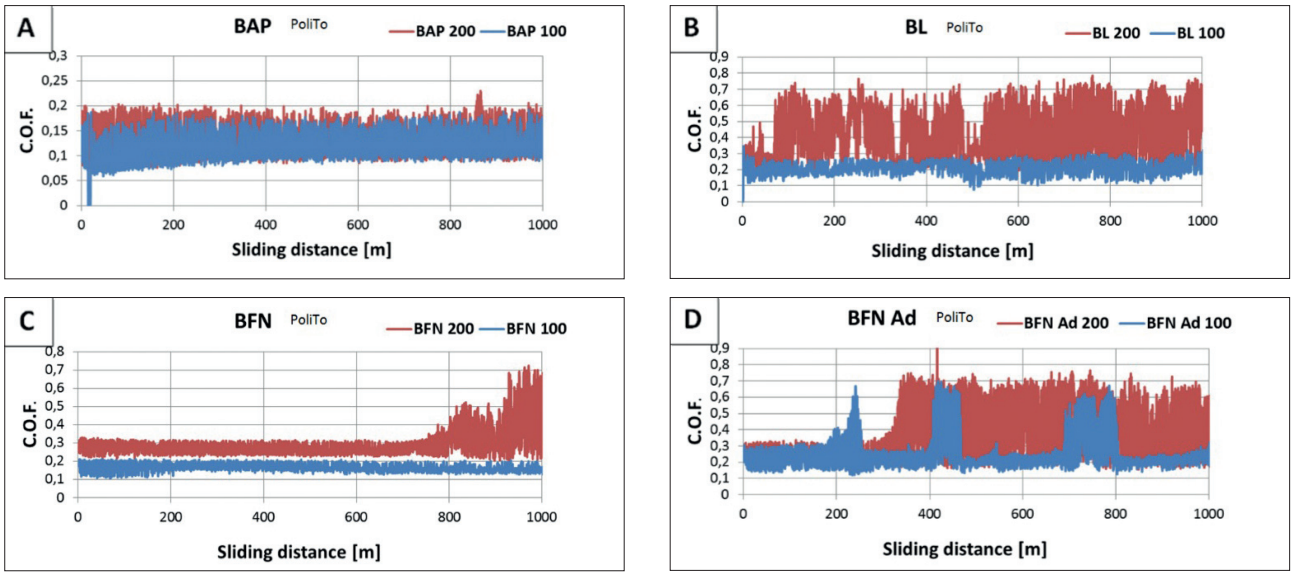

Fig. 3 - Coefficient of friction (COF) vs. sliding distance A) Balinit Alcorna Pro®; B) Balinit Lumena®; C) Balinit Futura Nano®; D) Balinit Futura Nano Advance®

The mass losses recorded on the pin and on the disk materials were measured using an analytic balance with accuracy to 0.001 g. Figure 4 shows the average mass loss of the pins for test performed after a sliding distance of 1.000 m and 2.000 m and the average mass loss of disks after a sliding distance of 2.000 m. These results show again that BAP was the coating grade which achieved the highest benefits from Laser Surface Texturing. This is measured by the fact that the pin wear, i.e. the material transferred from the pin (that resembles the workpiece) to the disk (that resembles the die) was limited. In terms of disc wear apart from BL (Balinit Lumena) all coating grades were unaffected. On the contrary, the BL was the thickest coating of the set and in this case LST introduced embrittlement which causes severe coating delamination. This provided either coating heavy damages and massive debris formations (weight losses in BL100) or steep increase in coating roughness acting as abrasive bits on the counterpart pin, thus enhancing material transfer (weight gains in BL200).

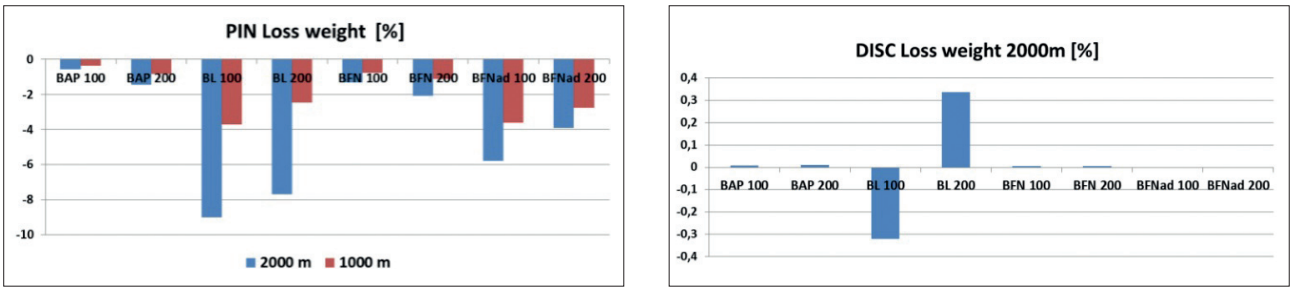

Fig. 4 - Average mass loss of the pins

Along the test the temperature of the pin tip was monitored (Figure 5). Although very limited temperature increases were recorded, also according to this measurement the LST BAP was the coating grade giving the better behaviour in terms both of maximum temperature achieved and of regularity of the temperature increase.

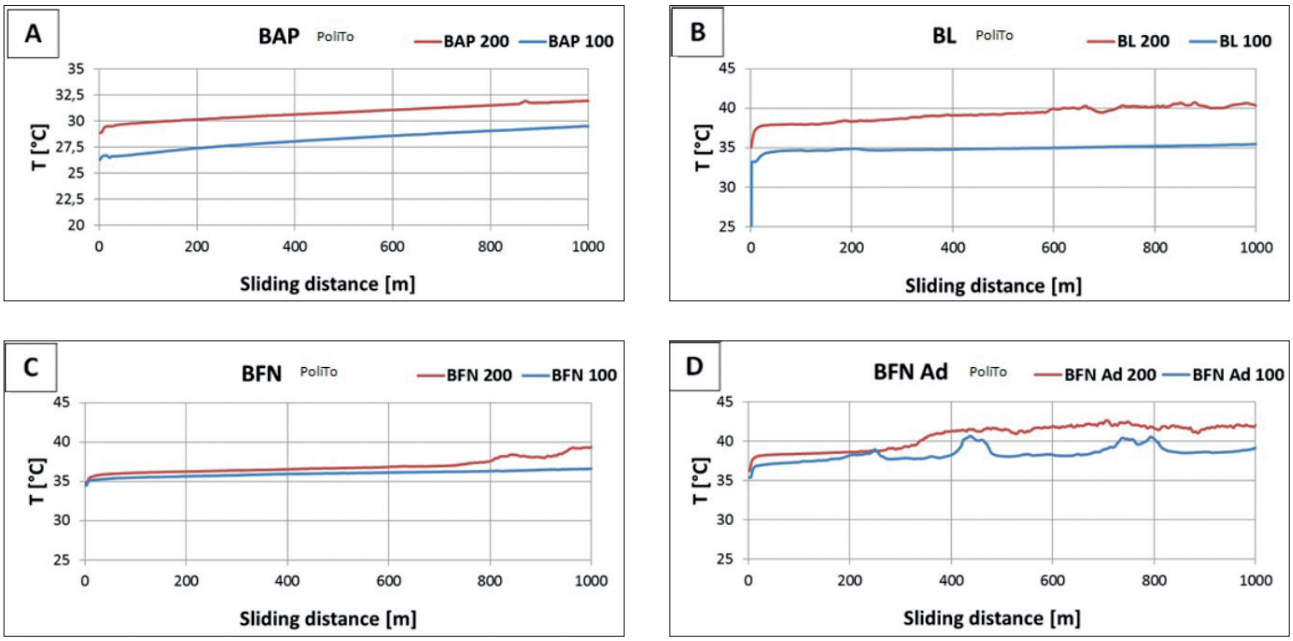

Fig. 5 - Temperature vs. sliding distance distance for different diameters:
A) Balinit Alcorna Pro®; B) Balinit Lumena®; C) Balinit Futura Nano®; D) Balinit Futura Nano Advance®.

To deepen the mechanisms which provided the behaviour for the different coatings SEM investigations along the wear track were performed. Figures 6 and 7 show some details of the tracks where transfer of steel from the counterpart pin to the LST coated disk occurred. The transfer was heavily present on BL, BFN and BFN Ad. LST samples, whereas it was very limited on BAP LST samples. The transferred steel was preferentially located around the dimples. In some cases the dimples are totally covered by steel (Figure 7 B2,C2 and D2). The transfer of steel was confirmed through EDS analysis. According to this investigation the combination of Balinit AlCRONA coating and of Laser Surface texturing was particularly positive in terms of limiting material transfer from the counterpart pin to the disk samples. On the contrary the other coatings performance in the wear tests were not positively influenced by LST.

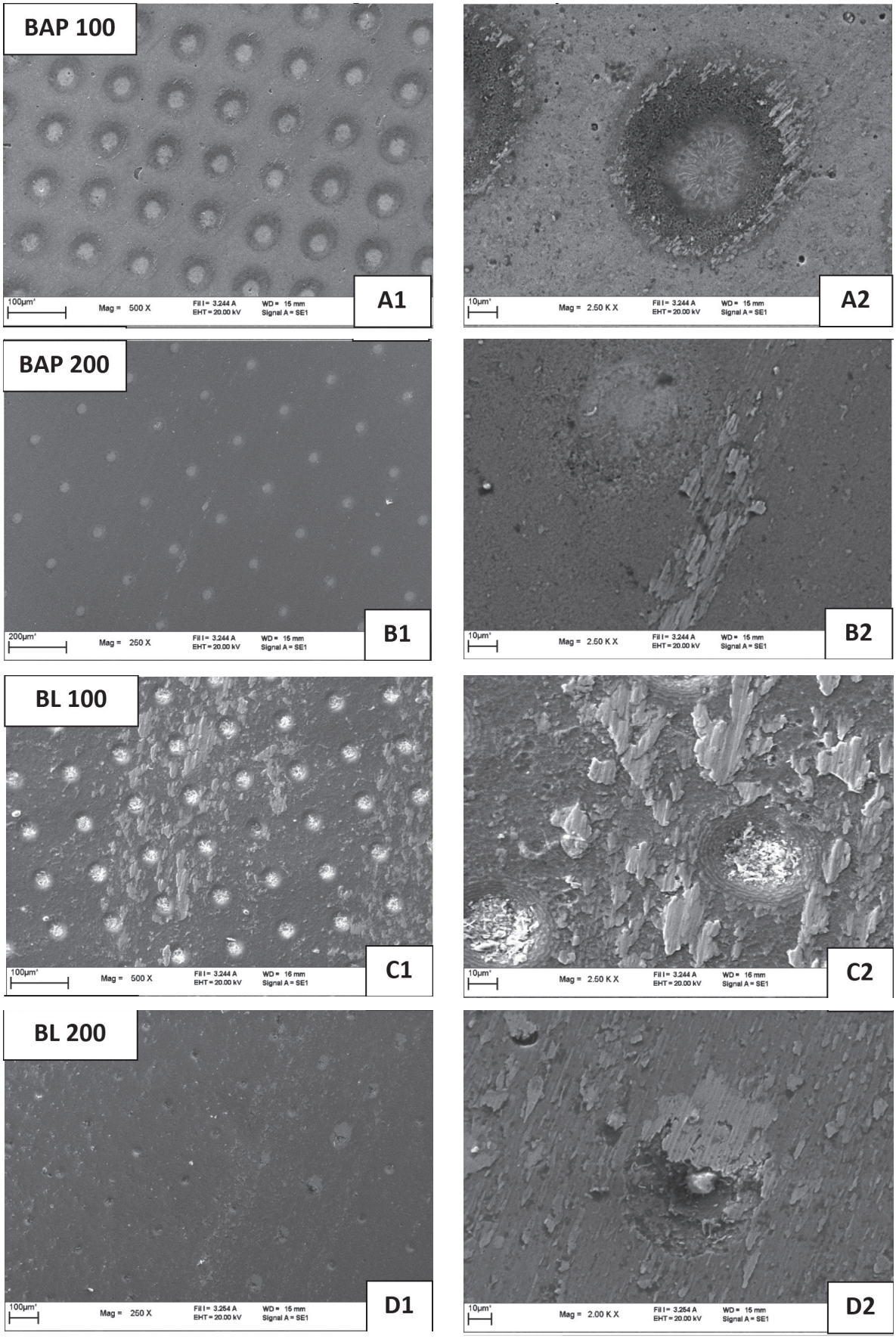

Fig. 6 - SEM images at different magnifications of tracks on the sample after the wear test performed on A) BAP 100 B) BAP 200; C) BL 100; D) BL 200.

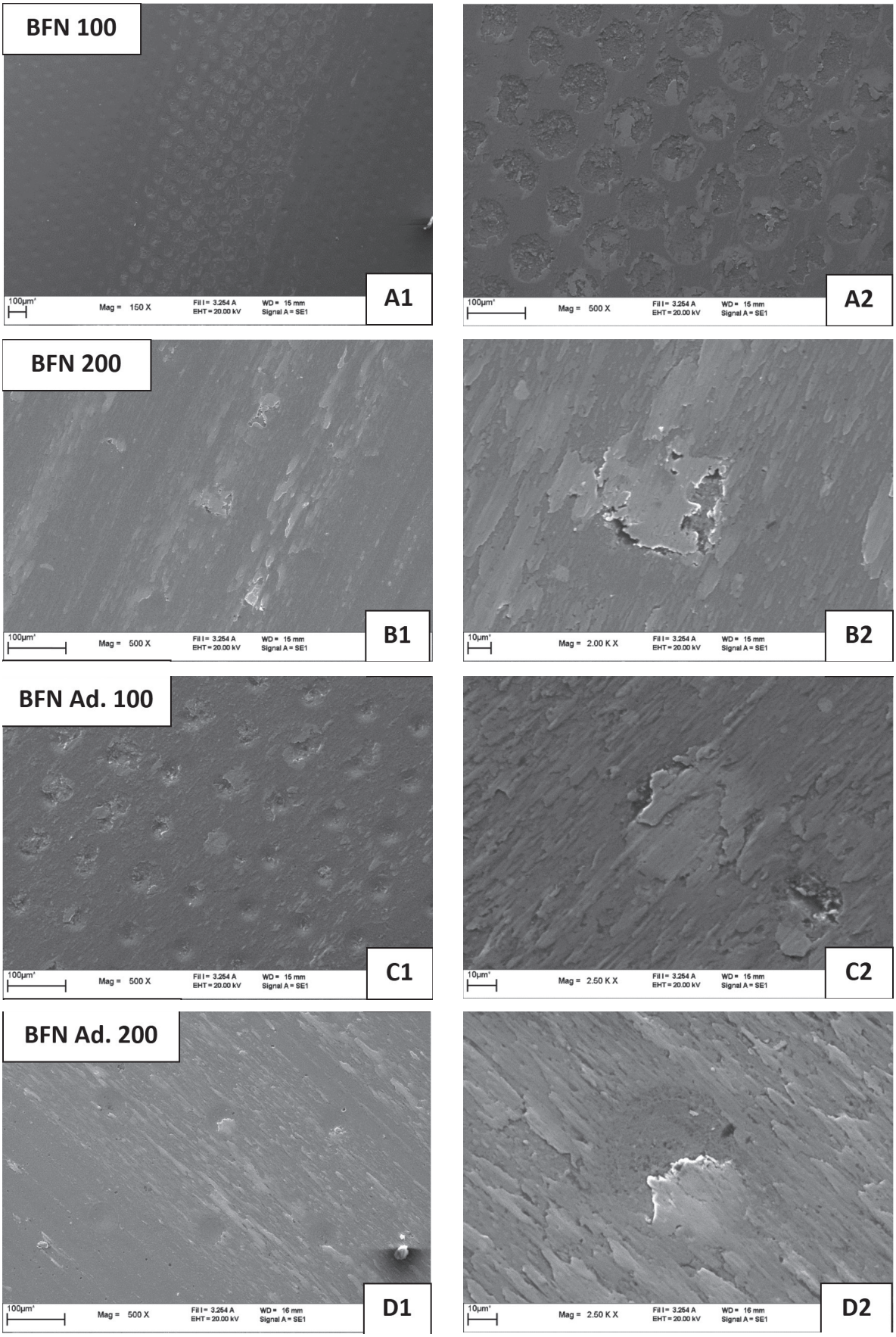

Fig. 7 - SEM images at different magnifications of tracks on the samples after the wear test performed on A) BFN 100; B) BFN 200; C) BFN Ad 100; D) BFN Ad 200.

The explanation for the benefits achieved in wear tests by using LST BAP lie in the capacity of the dimples structure generated on this coating to finely distribute and entrap the lubricant film, thus limiting the wear actions. The quality of dimples obtained on such coating is indeed higher than in other coatings and optimal laser coating interaction achieved allowed to obtain very regular dimples. This coating is based on AlCrN system which is a very thermally stable and inert material. This can be helpful both during dimple formation and during the test (service) duration. This promising behaviour of LST BAP was also confirmed in an additional although preliminary study on wettability of LST coatings, where a clear benefit in wettability towards lubricant oil was recorded for this coating in comparison with other LST coatings.

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CONCLUSIONS
A study on laser surface texturing of four different coating grades was performed. Dimple structures characterised by different geometrical features were machined on coated samples through a nanoseconds fibre laser system. Structural properties of coatings were assessed. Texturing was investigated mainly in terms of wear in a test where a stainless steel pin was used. This unusual selection was done to resemble the final application of the study, i.e. cold forming of stainless steel sheets. In this test plastic deformation of the pin indeed occur, but the most interesting information recorded was the tendency of coated and LST coated surfaces (i.e. the die material) to give adhesion with the counterpart stainless steel (i.e. the workpiece material). Among the studied coating grades the AlCrN coating system subjected to Laser Surface Texturing was the most promising one. In this case, stainless steel transfer was highly limited, regular coefficient of friction and very limited surface temperature increases were recorded. As for the other coatings, the dimples structure generated on them was not able to give any improvement in terms of wear performance, even providing massive stainless steel transfer into the generated dimples with a consequent difficulty in sliding of the counterpart material on these surfaces.

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