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TRIBOLOGICAL BEHAVIOR OF THERMAL SPRAY COATINGS, DEPOSITED BY HVOF AND APS TECHNIQUES, AND COMPOSITE ELECTRODEPOSITS NI/SiC AT BOTH ROOM TEMPERATURE AND 300°C.

A.Lanzutti¹, M. Lekka¹, E. Marin¹, L.Fedrizzi¹

¹ Università di Udine, Dipartimento di Scienze e Tecnologie Chimiche, Via del Cottonificio 108, 33100 Udine Italy,
alex.lanzutti@uniud.it

Abstract: Both the thermal spray and the electroplating coatings are widely used because of their high wear resistance combined with good corrosion resistance. In particular the addition of both micro particles or nano-particles to the electrodeposited coatings could lead to an increase of the mechanical properties, caused by the change of the coating microstructure.

The thermal spray coatings were deposited following industrial standards procedures, while the Ni/SiC composite coatings were produced at laboratory scale using both micro- and nano-sized ceramic particles. All the produced coatings were characterized regarding their microstructure, mechanical properties and the wear resistance. The tribological properties were analyzed using a tribometer under ball on disk configuration at both room temperature and 300 °C.

The results showed that the cermet thermal spray coatings have a high wear resistance, while the Ni nano-composite showed good anti wear properties compared to the harder ceramic/cermet coatings deposited by thermal spray technique.

Keywords: thermal spray coatings, nano-composite electrodeposits, Ni/SiC, micro-composite electrodeposits, HVOF, APS, dry sliding, 300°C).

1. INTRODUCTION

The thermal spray coatings are widely used for many industrial applications [1-13] because of the possibility to deposit different type of materials, ranging from different metal alloys to ceramics, and their technological properties, in particular the high wear resistance even if they are used also as corrosion barriers at both high temperature degradation or wet corrosion.

The thermal spray coatings are mainly used for high temperature applications (oxidation resistance or fused salts resistance). Usually these types of coatings are deposited with the addition of rare earths in order to inhibit the oxidative degradation processes [1-3]. Some technological processes are used to reduce the porosity of the coating and thus increase both mechanical properties and the barrier effect to oxidative environments. Sidhu et al [2]

have found that the laser remelting process increase both the mechanical properties and the oxidation resistance and leaves only a small amount of porosity to the coating (<1%). Sigh et al [3], instead, showed that the NiCr (80/20) has a good resistance to hot corrosion in molten salts at 900°C.

The literature about NiCr wear performance is scarce. Usually this kind of coating is used as bond coat for ceramic or cermet coatings in order to promote the adhesion of the deposited material to the substrate [4-6]. The NiCr 80/20 alloy is also used as metal matrix to produce composite coatings reinforced with carbides [7-9]. The WC-Co coatings, instead, were widely used and analysed by many research teams [7-12]. The interest about these types of coatings is related to their high mechanical properties that leads to high wear resistance of the coated system. Fedrizzi et al [7] performed some tribo-corrosion tests on cermet

coatings and observed their good wear resistance, related to their high hardness, but low corrosion resistance, in wet wear tests. This behaviour is related to the low toughness that promote the crack enucleations and thus the permeation of corrosion media that enhances the undermining corrosion of the coating. Toma et al [8] showed that the addition of Cr to metal matrix increased the abrasion and corrosion resistance. Fedrizzi et al [9] showed that these type of coatings can be used as an alternative to hard chromium and their performance are also increased if are used nano-sized powders. Murthy et al [11] showed that the coatings grinded have an higher wear resistance because of the production of an hard layer on surface. Other researcher [12] found out that the decrease in powder size increase the coating performance because of both reduction of the porosity and increase of the mechanical properties. This enhances both corrosion and wears protection.

The ceramic coating Cr_2O_3 was subjected to numerous studies in the area of both wear and corrosion protection. It was shown by Ahn et al that, in the reciprocating wear tests, the wear mechanism is a plastic deformation of wear debris that influence both the friction and wear behavior. This is related to a formation of CrO_2 layer under hertzian loads [4]. Bolelli et al [5] performed wear tests at room temperature on different plasma spray ceramic coatings. He found that the Cr_2O_3 coating is the hardest and most anisotropic coating with high abrasion resistance, as confirmed by Leivo et al [6], while in sliding condition the material forms a compact tribofilm. The high temperature data for this type of coatings is scarce.

The Ni based electrodeposits are widely used as both corrosion/wear barrier coatings in many applications ranging from high temperature applications to room temperature applications in both dry and wet conditions [14-21].

In this work composite coatings are also analyzed. The introduction of reinforcing particles is aimed to enhance both the wear and corrosion properties, in particular if the nano-particles are embedded to the metal matrix they produce a nano-structured microstructure.

Garcia et al [14] showed that the increase of wear properties at room temperature for micro-composite Ni/SiC coatings is a function of particle size. In particular the decrease of particle size leads to an increase of anti-wear properties.

The research group of Zimmermann et al [15-16] observed that the addition of sub-microsized particles to the coating leads to an increase of both mechanical strength and toughness, if the reinforcing content is below the 2 wt%. Above the 2wt% they showed that some particles' coalescence

is possible, during the deposition, leading to coating embrittlement. They tried to add nano-particles to the coating and observed a notable increase of the mechanical properties.

Benea et al [17-18] in many works demonstrate that the addition of SiC nano particles, in Ni matrix, leads to an increase of the wear properties and they calculated the relation between the microstructure of the coating and the wear performance.

The wear properties of both micro composite and nano composite Ni/SiC coatings at high temperature were investigated by Lekka et al [19]. They found an increase of anti-wear properties of the composite coating at both room temperature test and 300°C compared to the pure Ni coating.

This work aimed to compare the wear performance of the most used thermal spray coatings with the anti-wear properties of the Ni composite coating, highlighting the important properties of the composite coatings produced with a simple and cheaper technique compared to the thermal spray process.

2. EXPERIMENTAL

2.1 Samples production

For all types of the deposits ASTM 387 F22 steel plates (7×10 cm) and discs (d=5 cm) have been used as substrates (chemical composition in Table 1).

Table 1: Chemical composition of steel substrate ASTM 387 F22

C	Si	Mn	P	Cr	Mo	Fe
0.11	0.31	0.5	0.025	2.2	0.9	Bal.

The thermal spray coatings have been deposited using industrial procedures. The deposited coatings were: NiCr 80/20 and NiCr 80/20 + Cr_2O_3 deposited by APS (Air Plasma Spray) technique and WC CoCr 18/4 deposited by HVOF technique.

Regarding the Ni matrix coatings, three types of deposits have been prepared: pure Ni (to be used as reference). Ni containing microparticles of SiC and Ni containing nanoparticles of SiC. The electroplating bath used was a high speed nickel sulfamate plating bath having the following composition: 500 g/l $\text{Ni}(\text{SO } 3\text{NH}_2)_2 \cdot 4\text{H}_2\text{O}$, 20 g/l $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, 25 g/l H_3BO_3 , 1 ml/l surfactant $(\text{CH}_3(\text{CH}_2)_{11}\text{OSO}_3\text{Na})$ based industrial product. The deposition was carried out using a galvanic pilot plant (12 l plating tank) under galvanostatic control at 4 A/dm², 50 °C, under continuous mechanical stirring. The deposition time was 2.5h in order to obtain 70–80 µm thick deposits. For the production

of the composite coatings 20g/l of micro- or nano-powders were added into the electroplating bath, dispersed using ultrasounds (200W, 24kHz) for 30min and then maintained in suspension under continuous mechanical stirring during the electrodeposition. The micro-particles have a mean dimension of 2µm and a very irregular and sharp shape, while the nano-particles have a mean diameter of 45 nm [19].

2.2 Samples characterization

The specimens characterization includes microstructure, chemical composition, microhardness, wear resistance at both room temperature and 300°C and corrosion resistance in two different environments.

The microstructure of the specimens have been analysed by SEM (Zeiss Evo-40) + EDXS (Oxford instruments INCA) in cross section. Both the SiC content and the coatings' porosity were calculated using an image analysis software [13]. For nano composite coating The SiC content was measured through the measurements of RF GDOES (HR-Profile, Horiba Jobin Yvon), calibrated using 28 CRM (Certified Reference Material) samples. The system was set up using an Ar pressure of 650Pa and a applied power of 50W. The micro-composite coating were not analysed by the GDOES because of some issues related to the plasma erosion of the reinforcing particles [21].

Micro-hardness measurements ($HV_{0,3}$) have been performed on cross section of the specimens.

Wear tests have been performed using a CETR UMT tribometer in a ball-on-disc configuration at both room temperature and at 300 °C. The testing parameters are summarized in Table 2. The volume loss has been evaluated using a stylus profilometer (DEKTAK 150 Veeco). The wear rate K [10^{-6} mm³/Nm] has been calculated using the equation described in [22].

Table 2: Wear test parameters.

Counter material	WC sphere (d 9.5mm)
Applied load	70N
Test radius	18mm
Rotation speed	300rpm
Sliding speed	0.565 m/s
Test duration	60 min

3. EXPERIMENTAL RESULTS

3.1 Microstructural characterization

In Fig. 1 is shown the microstructure of the steel substrate.

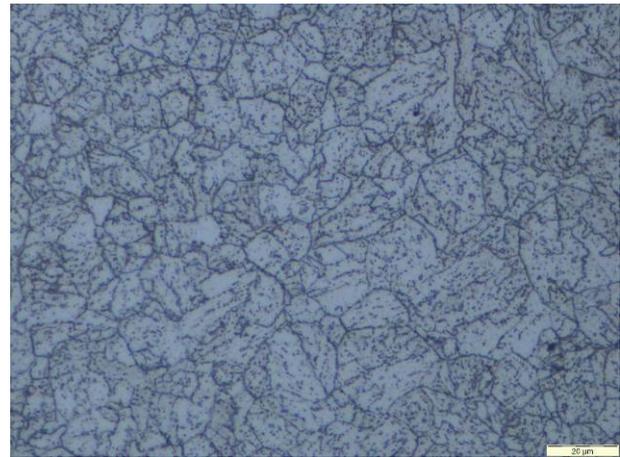
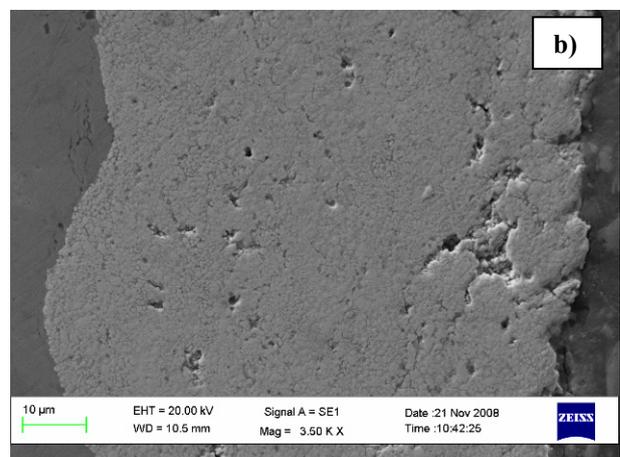
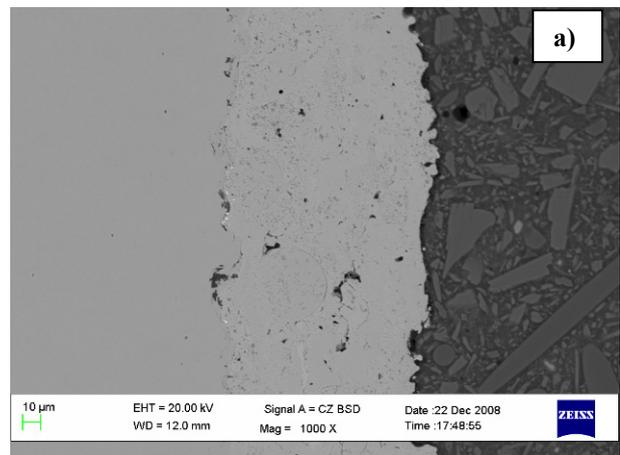


Fig.1: Microstructure of gr. 22 steel.

The Gr 22 steel presents a ferritic microstructure with some carbides precipitated in the metal matrix, that leads to the high creep strength of material. The carbides are mainly produced by Cr and Mo. The hardness of the material is about $180 \pm 20 HV_{0,3}$ and the ferritic grain size is about $45 \pm 15 \mu m$.

In Fig.2 are shown the SEM micrographs obtained for Thermal spray coatings and the relative data acquired by mechanical characterization and image analysis. In Tab. 3 the thermal spray coatings' properties are listed.



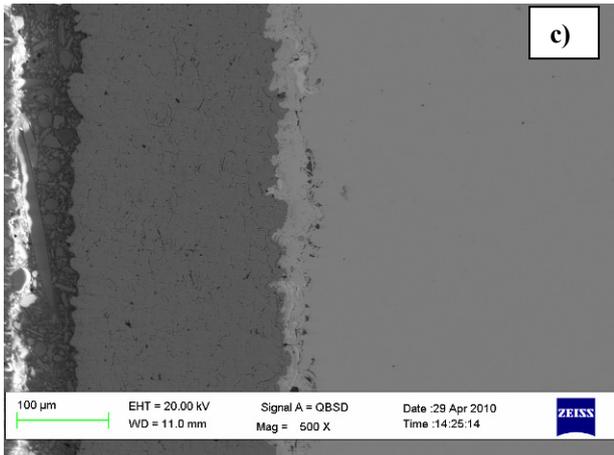


Figure 2: SEM images and microstructural characterization of thermal spray coatings: a) NiCr 80/20, b) WC CoCr 18/4 and c) NiCr 80/20+ Cr₂O₃.

Table 3: Results of thermal spray coatings' characterization.

Coating	Thickness [µm]	Porosity vol.%	Hardness HV _{0.3}
NiCr	98±16	6.5	359±18
WCCoCr	105±15	3.45	1027±21
NiCr+Cr ₂ O ₃	(38+187) ± 25	5.5+10.1	(341+1118) ±24

As can be observed, the three types of thermal spray coatings present different thickness and porosity. The porosity, acquired by image analysis, is higher for the coatings deposited by APS technique compared to the HVOF deposits. This difference could be related to both powders size and impact velocity, that is lower in APS technique with respect to HVOF. Indeed, the difference in kinetic energy of the molten powders, that is higher in HVOF technique, leads to a different density on deposited coating. The hardness acquired is associated to the material deposited and the values acquired are similar to data available in scientific literature for thermal spray coatings [1-13].

The SEM micrographs obtained on cross section of Ni/SiC composite coatings previously etched (acetic acid: nitric acid 1:1) are shown in Fig.3. In Tab. 4 are listed the electrodeposited coatings' properties.

Table 4: Results of Ni/SiC electrodeposits characterization.

Coating	Thickness [µm]	SiC wt.%	Hardness HV _{0.3}
Ni	78±7	-	172±7
Ni/µSiC	73±8	0.8	247±8
Ni/nSiC	75 ± 5	0.15	270 ±9

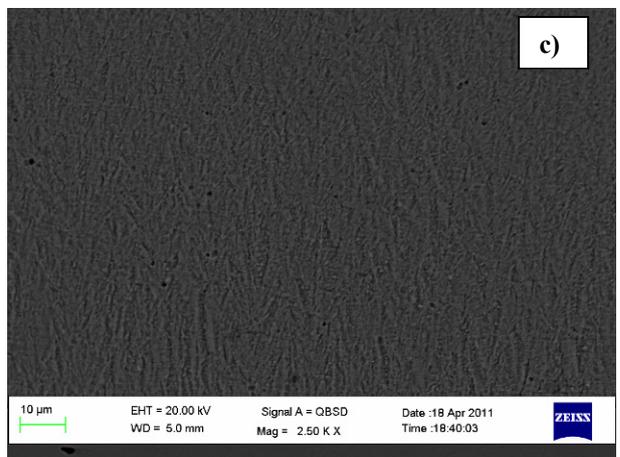
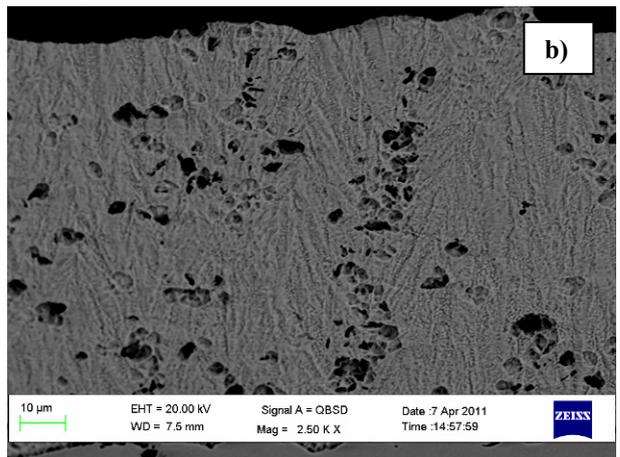
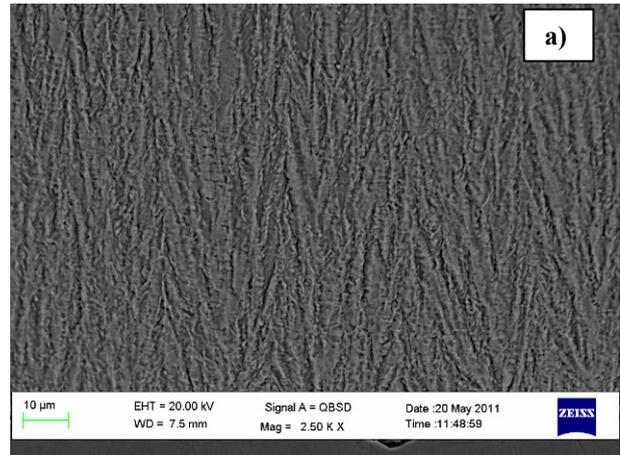


Figure 3: SEM images and microstructural characterization of electrodeposited coatings: a) pure Ni, b) Ni/µSiC and c) Ni/nSiC.

The microstructure of the electrodeposits is columnar. In the case of the pure Ni the metal columns are oriented along the direction of electrical fields. The addition of SiC micro-particles leads to a slight modification of the Ni columns orientation and size. On the other hand, the codeposition of SiC nano-particles leads to the formation of a fine grained deposit in which the Ni columns are not oriented. The addition of micro-particles leads to a microstructure columnar with a slight modification of orientation, caused probably

by the deviation of electrical field in proximity of ceramic particles that are in non-conductive material. On the contrary, the addition of nanoparticles gives a grain refinement and a multi-orientation of columns. The SiC amount is higher in the micro-composite coatings compared to the nano-composite one. The addition of particles leads to a noticeable microhardness increase due to both the presence of the particles and the grain refinement.

All the analysed samples showed a surface Roughness Ra of about 0.5µm, obtained after the surface' grinding.

3.2 Tribological characterization

All the wear tracks obtained for both bare steel, thermal spray and composite electrodeposits at both room temperature and 300°C are shown in Fig. 4-6.

In Fig. 4 the top views of the wear tracks obtained for gr.22 steel are shown, tested at both room temperature and 300°C.

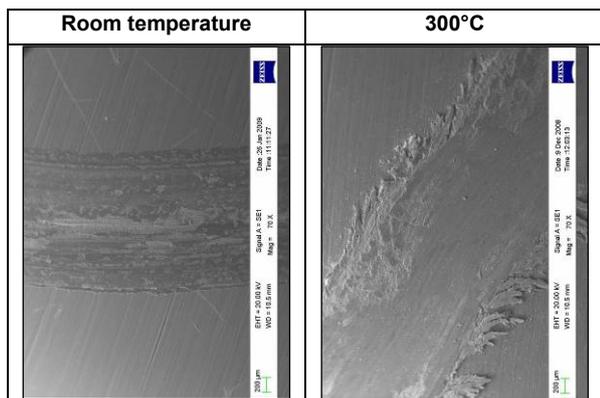


Figure 4: SEM images of the wear tracks obtained for the bare steel at both RT and 300°C.

The steel is subjected to tribooxidative wear at both room temperature and 300°C. At room temperature the oxide produced is adherent to metal substrate and very homogeneous. At 300°C the oxide produced is concentrated on the wear track' sides. This phenomenon is related to the loss of mechanical properties of the substrates that permits the counter material to destroy the oxide layer, that is thus deposited on the sides of the wear track. At 300°C are highlighted the debris of both oxide and steel along the borders of wear track.

In Fig. 5 the top views of the wear tracks obtained for thermal spray coatings are shown, tested at both room temperature and 300°C.

For the metal matrix deposits the degradation mechanism is a tribooxidation at both room temperature and 300°C, which intensity is varying in function of the coating material.

In particular the thermal spray coatings showed also other degradation mechanisms which are related to their microstructure.

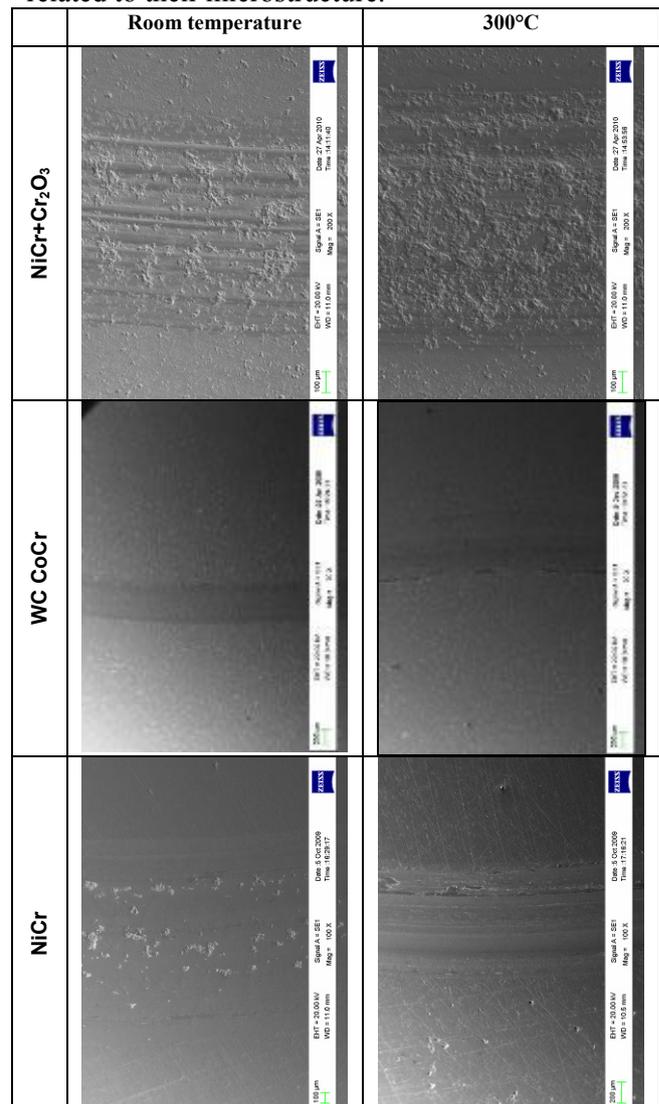


Figure 5: SEM images of the wear tracks obtained for the thermal spray coatings at both RT and 300°C.

The Ni/Cr showed a material detachment originated by contact fatigue phenomenon, aggravated by its porosity. At high temperature tests the detachment is decreased due to a possible hardness decrease of the material which allowed the sealing of porosity, thus reducing the contact fatigue failure. The cermet coatings were all subjected to tribooxidation of metal matrix, more intense at high temperature. For the ceramic material (Cr₂O₃) the degradation mechanism at room temperature is similar to the NiCr coating while at high temperature test the degradation becomes more intense. This is caused by a phase change of chromium oxide which enhances the wear ratio and reduces at the same time the friction coefficient [4].

In Fig. 6 are shown the top views of the wear tracks obtained for Ni/SiC electrodeposits tested at both room temperature and 300°C.

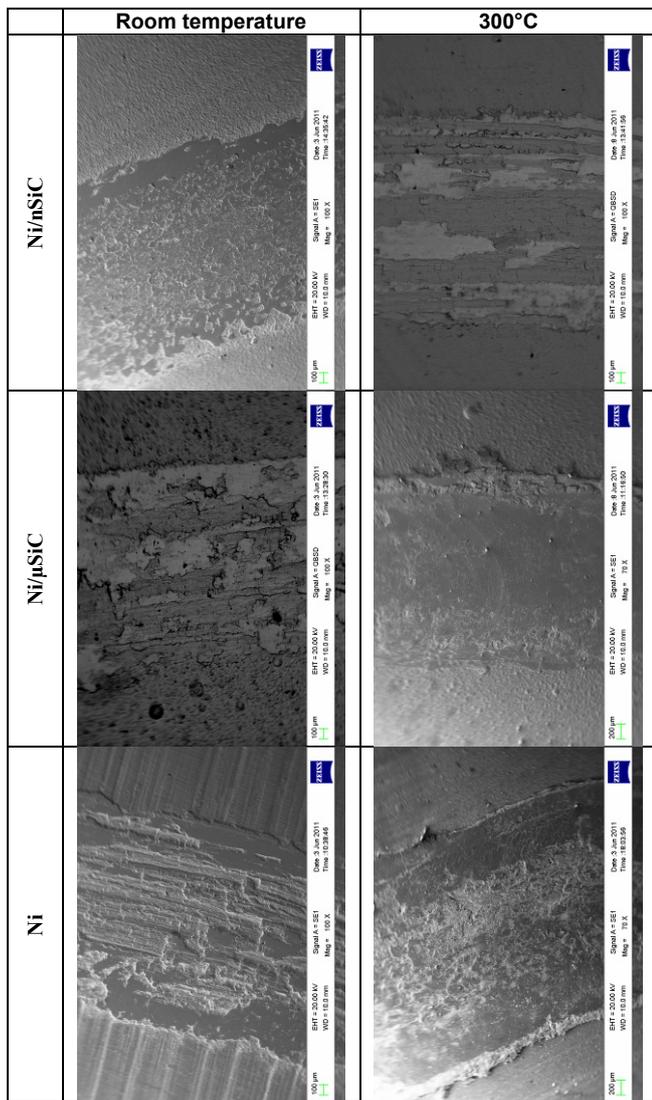


Figure 6: SEM images of the wear tracks obtained for the Ni/SiC deposits at both RT and 300°C.

The Ni electrodeposits showed, at room temperature, a tribooxidation with a descaling of oxide which forms a third body between the counter material and the wear track, thus leading to the formation of secondary tracks related to abrasive wear. At high temperature the coatings showed a strong tribooxidation. By EDXS analysis on pure Ni coatings, it was detected that during the wear test the steel substrate was reached. For the composite Ni/SiC coatings it seems that the oxide produced is more adherent to steel substrate at high temperature. Probably both the grain refinement and the presence of ceramic particles are linking the oxide to the metal matrix. It seems that some counter-material was transferred to the coating surface, due to a slight adhesion phenomena.

The wear rate of all tested coatings at both room temperature and 300°C are shown in Fig. 7.

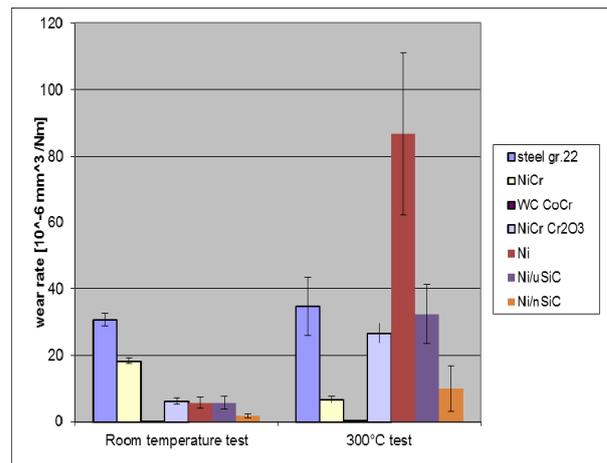


Figure 7: wear rates graph at both room temperature and 300°C for all the coatings tested.

All the coatings protect the steel substrate during the test, except for the pure Ni coating that showed, at 300°C, the highest wear rate. It is possible to observe that the cermet coating has the lowest wear rate compared to the other coatings at both test temperatures. This is related to the high amount of WC which is bonded by a metal matrix that has high oxidation resistance at the test temperatures. For this coating the wear resistance is associated to the carbide component and the particles binding is related to the metal matrix which has a high toughness. On the other hand, the NiCr coating showed a lower wear rate at high temperature tests, compared to the room temperature one, and this could be related to both high oxidation resistance of the material, that contains a high amount of Cr, and to the reduction of material detachment that consequently reduces the abrasive phenomena. The ceramic coating (Cr₂O₃) showed a high wear rate at 300°C tests due to phase change of chromium oxide under tribological contact.

The electrodeposits showed good wear resistance at room temperature, higher for the nano-composite coating. This reduction in wear rate could be related to the grain refinement of microstructure of the metal matrix, which increases also the mechanical properties of the coating. This effect is not visible in the micro-composite coatings because the reinforcement particles are usually detached from the metal matrix leading to intensive abrasive wear caused by hard particle third body contact between counter material and surface of the specimen. At high temperature the mechanical behaviour of the coating is reduced, probably because of the hardness decrease. In this case the pure Ni coating is completely removed while the micro composite coating showed a better wear resistance, compared to the pure Ni one, but the wear rate values were still higher than the thermal spray coatings wear rates. The higher wear

resistance of the nano-composite coating at 300°C is probably related to the higher mechanical properties of the metal matrix compared to the other electrodeposits.

In fig 8-9 the COF (Friction Coefficients of the tested materials) are shown.

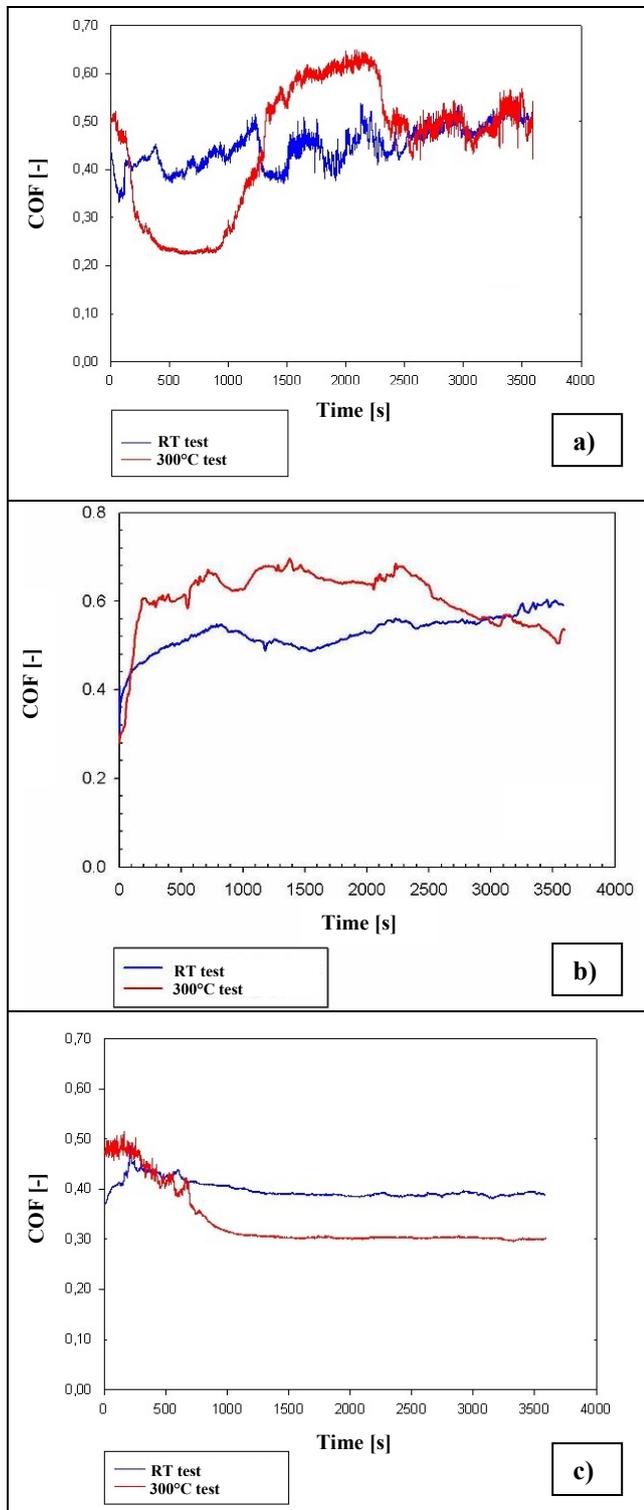


Fig.8: COF graphs at both room temperature and 300°C for the thermal spray coatings: a) NiCr 80/20, b) Wc Co Cr (18/4), c) NiCr 80/20+ Cr₂O₃.

For all the test performed on thermal spray coatings, it is possible to observe that the COF values, at the end of the test, are comparable between the tests performed at different temperature, apart the ceramic coating that showed a lower COF at high temperature due to the change phase of ceramic oxide under hertzian loads. The NiCr coatings showed a noisy COF graph because of the coating material detachment that produced abrasive particles that dissipated more energy, required to move the particles in the hertzian system. At high temperature there is a start at low COF and, at regime, it reached the same values of the test at room temperature. Probably during the start of the test the surface of the sample was covered by a oxide layer produced during the heat up of the system. The presence of oxide decreased the surface energy in proximity of the hertzian contact of the two materials, reducing the friction coefficient. When the oxide was broken the contact between the two materials was between the WC and the Ni/Cr slightly oxidized.

For the WC-CoCr coating the COF are slightly different and this is caused mainly by the number of third body particles produced during the test. Indeed is possible that at high temperature the amount of abrasive particles, that are taking part to the hertzian system, are higher due to the intense trioxidation that cause probably a high amount of descaled oxide. At the end of the test part of the particles are evacuated from the wear track reaching a COF value comparable with the room temperature test.

The COF acquired from the test performed at 300°C is lower compared to the value acquired at room temperature test. This behaviour is related to the change phase of ceramic oxide that decreased the contact energy and thus the COF. The friction coefficient values are higher at the start of the test because of possible partial fragmentation of the material caused by brittle contact between the counter material and the coating. This leads to have an high amount of third body particles that increase the COF value, at the beginning, that is decreasing, during the test, because of particle' evacuation form the wear track caused by the relative motion of the two materials.

The COF acquired for the Ni/SiC electrodeposits is lower compared to the pure Ni electrodeposit. This could be related to both different mechanical properties of the composite material respect to the pure Ni and possible interactions of SiC particles with counter material that could lower the surface energy and interaction of the 2 surfaces.

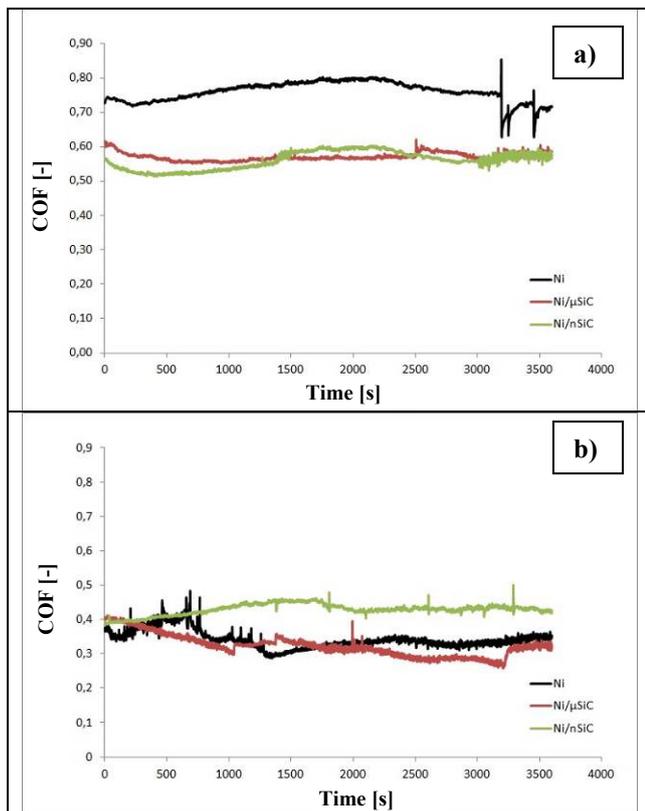


Fig.9: COF graphs at both room temperature and 300°C for the Ni/SiC composite coatings: a) Room temperature test, b) 300°C.

At high temperature the COF graphs are very similar and this behaviour could be related to the change of contact, compared with room temperature test, that is between Ni oxide and the WC sphere, instead of Ni slightly oxidized and WC.

4. CONCLUSIONS

In this work different type of coatings have been analysed deposited either by thermal spray techniques or by electrodeposition. The coatings deposited by thermal spray are: NiCr (APS), WC CoCr (HVOF) and NiCr+Cr₂O₃ (APS). The electrodeposits are Ni/SiC coatings with nano- or micro-sized particles embedded in metal matrix.

The analysed coatings showed different microstructure that depends on both deposited material and deposition technique.

Regarding the wear properties, The steel substrate showed the worst wear resistance at both room temperature and 300°C. This behaviour is related to the low mechanical properties of this steel, that are decreasing as the temperature increases.

All the tested metal matrix coatings underwent tribooxidation, that was increased at high temperature test. The tribooxidation behaviour depends on metal oxidation resistance. The ceramic

coating was subjected to an intensive material detachment, caused mainly by the high interconnected porosity of the thermal sprayed coating. The detachment increased in function of temperature because the ceramic oxide changed phase under the hertzian loads. For all the metal matrix coatings was present a third body abrasion caused mainly by both oxide descaling and ceramic reinforcement detachment from the metal matrix.

Observing the wear rates, the WC CoCr coating showed the highest wear resistance at both room temperature and 300°C. This behaviour is related to the microstructure of the deposit: the reinforcing particles (WC) give high hardness also at high temperature and the metal matrix (CoCr) increases the toughness of the coating and acts as binder for the reinforcing particles. The electrodeposits Ni/nSiC showed a wear behaviour that is comparable with the WC CoCr one. For the nanocomposite electrodeposits the synergy of both grain refinement and nano-particles embedding leads to an increase of hardness at both room temperature and 300°C. This effect probably enhances the wear resistance of the Ni metal matrix that is subjected to hertzian loads.

The COF values are strongly dependent on the material analysed but it was observed, for thermal spray coating, similar COF values between the room temperature test and the 300°C tests. The ceramic coating showed the lowest COF values at high temperature caused mainly by the production of brittle CrO₂ phase. The electrodeposits showed some differences in the COF values between the high temperature tests and the room temperature tests caused mainly by the change of hertzian contact from Ni slightly oxidized, at room temperature, to Ni strongly oxidized, at 300°C. At room temperature is visible a different in COF value between pure metal and composite coatings. This effect is related to the different mechanical properties of the coating and the possible interaction of reinforcing particles with the counter-material in the hertzian contact/motion.

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