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TRIBOLOGICAL BEHAVIOR OF 410L SS - SiCp COMPOSITES REINFORCED WITH SIC PARTICLES FUNCTIONALIZED BY Ti-Ni COATING LAYERS

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Abstract: *This study is concern with the tribological behavior of 410L SS - SiCp composites reinforced with SiC particles functionalized by Ti-Ni coating layers. The 410L SS composites were produced by hot-pressing technique. For comparison purposes the 410L SS alloy and the uncoated SiCp reinforced composite - 410L SS/SiC, were also tested. A reciprocating ball-on-plate tribometer was used to evaluate the tribological behavior. The produced materials were tested against a steel ball. The worn surfaces were analyzed through SEM/EDS. The results indicated that the combination of PVD and electrocoating is a feasible process for reactivity control between the reinforcement and matrix. The results show that using a titanium-nickel coating is helpful in improving the wear resistance of 410L SS composites.*

Key words: *SiCp, Stainless steel, MMCs, Wear, Friction.*

1. INTRODUCTION

410L stainless steel (SS) has been used due to its excellent mechanical properties in many applications (e.g. cutting tools, surgical tools, etc.), where high strength is required. However, its behavior is limited when there are parts in contact and in a relative motion, such in the case of cutting tools where the surface is exposed to sever conditions by a counterface material (fast movement, high temperature etc.) [1-3]. In order to improve the tribological performance of this steel several approaches have been used, e.g. using different thermal treatments in order to increase hardness or the development of metallic matrix composites (MMCs) [4–6].

It has been already demonstrated that incorporation of ceramic particles (e.g. SiC) into 410L stainless steel matrix will increase the hardness [4,7]. Till now, processing technologies such as casting or powder metallurgy (PM) were used to produce MMCs. PM technology has several advantages, such as superior control over microstructure, controlled levels of porosity, processing of materials with very high melting points etc. [4,8].

The reaction between the metal matrix and ceramic reinforcement has a great influence in the properties of the composites [9,10]. This reaction can be controlled by using different processing technologies (HP, SLM, LENS etc.), or changing the matrix or applying a surface treatment on the reinforcement [11].

Several processes were used to apply coatings, such as gaseous (e.g. chemical vapour deposition, physical vapour deposition - PVD etc.), solid (chemical solution deposition, electrochemical deposition etc.) and molten or semi-molten processes [1]. In the case of PVD, which is a gaseous process, an inert gas is ionized by creating a differential potential. The ionized gas is accelerated to target promoting a collision. During the collision, material is removed from the target and projected to a subtract forming the coating [12]. Generally, the PVD process is used to coat the surfaces of a components. To the knowledge of the authors, there are very few studies that use the PVD as a process to coat powders [13–16]. The devices used till now to coat powders are: Rotary Barrel Coating, Hexagonal Rotary Barrel Coating, Conical chamber with deflectors in inner wall and Vibratory plate [16–21].

The reactivity between SiC particles and 410L stainless steel alloy was studied in a previous study of the same authors, by using different sintering temperatures from 900 - 1180°C. It was demonstrated that the reaction between the SiC particles and 410L stainless steel alloy is highly dependent on the temperature. At 900°C no reaction zone was observed, while by increasing the temperature to 1180°C, the SiC particles completely dissolved. The optimal parameters to have a good reaction zone where the temperature 1100°C and the pressure 1200 MPa. In this case two zone were observed: FeSi phase (inner zone) and Fe_3Si (outer zone) [4].

In this paper the tribological behavior of 410L SS - SiCp composites reinforced with SiC particles functionalized by Ti-Ni coating layers was studied. The reactivity between SiCp and 410L stainless steel alloy was controlled by applying a coating layer of titanium and titanium-nickel on the SiCp. The results show that by using a titanium-nickel coating is helpful in improving the wear resistance of the 410L SS composites.

2. EXPERIMENTAL PROCEDURE

2.1 Materials

A martensitic stainless-steel powder 410L SS (Hoganas AB – Sweden) and silicon carbide particles (SiCp), were used to produce 410L SS/SiCp composites. Fig. 1 shows the SEM images of the 410L SS powder and SiC particles.

410L SS powder with a particle distribution of 45% lower than 45 μm and 1% above 150 μm, and a 1 weight percentage (wt.%) lubricant (Acrawax) was used as matrix. The chemical composition of 410L SS is presented in Table 1. SiC particles with a mean particle size of 118 μm were used as reinforcement.

Table 1

Chemical composition of 410L SS powder (wt. %).

Element	C	Cr	Si	O	N	Fe
(wt.%)	0.02	13	0.8	0.24	0.03	Bal.

2.2 Sputtering functionalization of SiC particles

SiCp particles were functionalized by Ti-Ni coating layer using PVD and electrocoating processes.

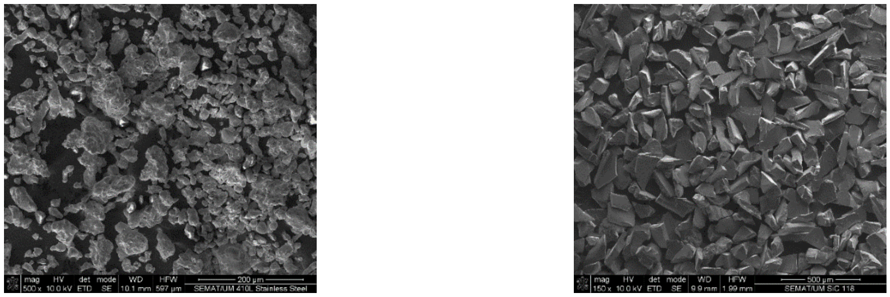
(i) Physical vapour deposition of Ti. The Ti coating was deposited on SiC particles surfaces using a PVD process. Fig. 2 shows the PVD-Rotary Barrel particles coating device. The main rotation axis (1) is connected to the motor (2) outside the deposition chamber (3), while (4) is Ti target and (5) the PVD-rotating cup/ barrel for powder deposition. To prevent the possible adhesion of the particles to the cup walls/corners a rotating arm (6) is attached to the main rotation axis which forms with (7) a knocking mechanism, while (8) is an attachment to (7) in case further weight is needed for the knocking mechanism. (9) is the bevel gears assembly which transfers the rotation to the PVD-rotary barrel particles coating device (5). The rotary cup/ barrel particles coating device has a novel design of the blades comparing to the ones presented in technical literature [20,21]. As a result, the PVD-rotary barrel blades (10) provide the necessary circulation to the powder (11), while the extension to the barrel wall (12) is to maintain the particles within the barrel during the process. To do the PVD coating, the SiC particles were placed into a PVD-rotating cup/ barrel that is inclined at 30°. The cup is open on the top, while radially in the inner part has blades. The cup is rotating around his axis during the deposition while the powder falls gradually from the blades (Fig. 2-e). The experimental conditions used for the PVD process are presented in Table 2.

Table 2

Experimental conditions of PVD (without sputter etching) of Ti coating on SiC particles.

Parameters	Values
Target dimensions (Ti)	965×140 mm
Powder mass (Substate)	3.5 g
Initial pressure	2.3×10^{-5} mbar
Deposition time	4800 s
Atmosphere	Argon – 20 sccm
Target current	7 A
Deposition temperature	91ºC
Target voltage	320 V
Depostion pressure	1.4×10^{-3} mbar
Substate Bias Voltage	80 V
Substate Current	0.306 A

ii). Electrocoating deposition of Ni. The Ni coating was deposited on SiC particles surfaces using an electrocoating deposition process. The process is schematically presented in Fig. 3 and the experimental conditions used for the electrocoating process are presented in Table 3.



(a) (b)

Fig. 1. SEM images of (a) 410L stainless steel powder and (b) SiC particles.

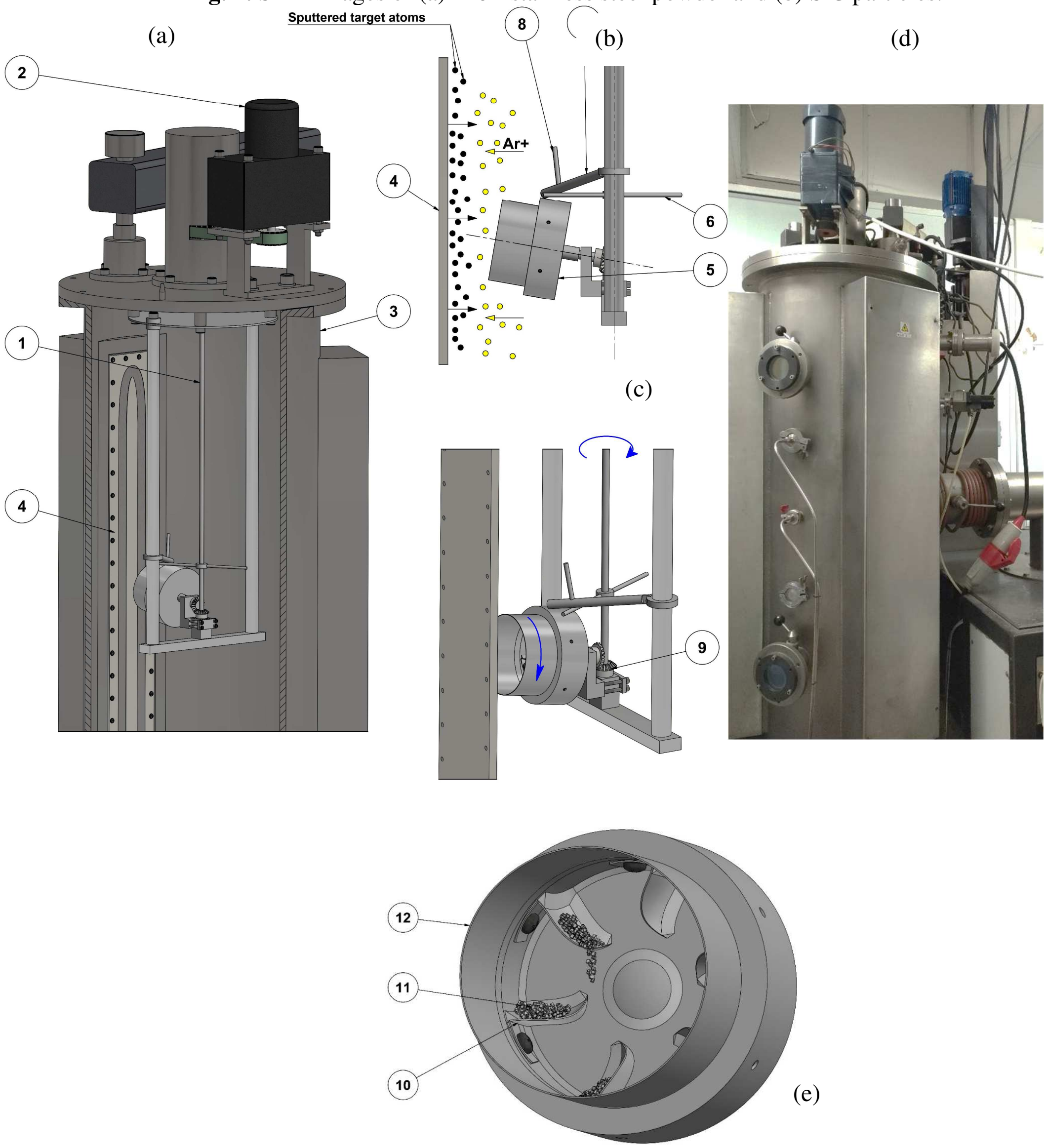


Fig. 2. The PVD-Rotary Barrel particles coating device, where:
a) the PVD chamber equipped with the rotary barrel particles coating device, b) and c) the PVD process during the rotation of the barrel, b) the actual PVD chamber, e) the rotary cup/ barrel particles coating device.

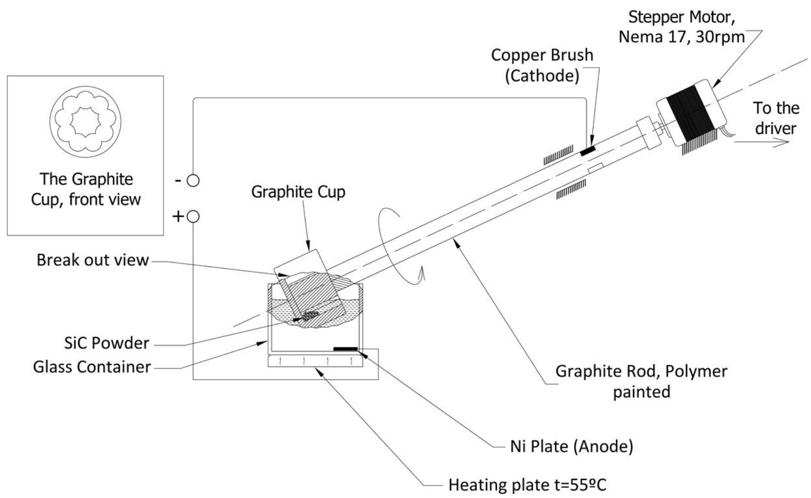


Fig. 3. Schematic representation of the electrocoating process.

Table 3

Experimental conditions used for the electrocoating process of Ni coating on SiC particles.

Parameters	Values
Voltage	2V
Time	30 min
Rotation	30 rpm
Temperature	55ºC

The SiC PVD coated powders are placed in a graphite cup/barrel which is immersed in electrolyte solution in a glass container. The copper brush is the cathode and the Ni plate is the anode. During the deposition the graphite cup/barrel is rotating to move the SiC particles.

2.3 Fabrication of 410L SS/SiCp composites

After functionalization of SiC particles, three types of 410L SS composites were produced through traditional hot pressing (HP) technique (Fig. 4): (i) 410L SS-matrix composite reinforced by uncoated SiC particles (named here 410L/SiC); (ii) 410L SS-matrix composite reinforced by Ti coated SiC particles (named here 410L/SiC-Ti); (iii) 410L SS-matrix composite reinforced by Ti/Ni coated SiC particles (named here hybrid composite - 410L/SiC-Ti-Ni).

410L stainless steel alloy was also produce through the same technology to serve as a baseline for all comparisons.

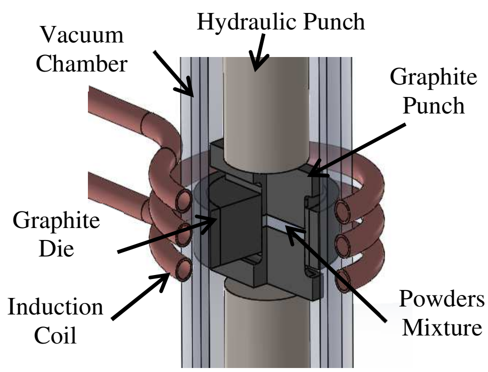


Fig. 4. Schematic representation of the HP apparatus [23].

The 410L alloy and 410L/SiC composite samples were produced by HP procedure in vacuum (10^{-2} mbar). After mixing the 410L SS powder and SiC particles inside a closed stainless-steel jar in argon (by using a rotation machine with a constant rotation speed of 40 rpm during 6 days), the mixture was placed inside a graphite die. As can be seen in Fig. 4 the die is then placed in a vacuum chamber, where it was compressed at required pressure 35 MPa at ~550°C started by around 1.3MPa before it heated up with a heating rate of 25°C/min. The sample was maintained at ~550°C and under the respective pressure during 10 min. Then, the samples were cooled inside the die till room temperature, in vacuum. More details regarding the employed HP method are provided elsewhere [4,22].

2.4 Details of the wear tests

Cylindrical test samples (10 mm diameter and 4 mm thick) were wet ground on SiC sandpapers down to P4000 and then polished using diamond paste (1µm). Samples were ultrasonically cleaned in isopropyl alcohol for 15 min and then in distilled water for 15 min. A reciprocating ball-on-plate tribometer (Bruker-UMT-2) was used to evaluate the wear behavior. As counter material was used a 10 mm diameter steel ball. The tribological parameters were 3 N normal load, at a frequency of 1Hz, and 3 mm of displacement, during 60 min. A set of five experiments was performed for each test condition. All tests were performed in laboratory environment (20 ± 2°C). The tribological tests were performed only in the case of 410L SS alloy; 410L/SiC composite and 410L/SiC-Ti-Ni hybrid composite.

3. RESULTS AND DISCUSSION

3.1 Morphological and chemical characterization of SiC particles with Ti PVD coating

Fig. 5 illustrates the SEM morphology (with different magnifications 250×, 2000× and 50 00×) of the SiC particles after applying the Ti PVD coating. To identify the composition of the applied coating, EDS analysis was performed in the marked zones presented in Fig. 5 (b) and (c) (Z0 to Z4). The EDS results are presented in Table 4.

Table 4

Chemical composition (wt.%) of the marked zones (Z0 to Z4) in Fig. 5.

	Z0	Z1	Z2	Z3	Z4
C	31.42	35.51	36.02	26.43	20.44
Si	66.8	62.31	57.34	66.98	37.63
Ti	1.78	2.19	6.64	4.8	29.41
O	0	0	0	1.79	12.52

The EDS analysis revealed the presence of Si and C coming from the SiC particles. In all tested zones was observed the presence of Ti, which proves that the SiC particles are covered by continuous Ti PVD coating. The O was detected only in zones Z3 and Z4. The presence of O can be explained based on the fact that titanium has a high affinity for this element in the interface of SiC and titanium (due to the fabrication process titanium may have residual oxide layers on their surface).

3.2 Interface reactivity

Fig. 6 shows the SEM image of a SiC particle after applying the Ti PVD coating and also the EDS analysis performed in two different zones: inside the SiC particle (zone B) and at the interface (zone A).

SEM images showed the continuous layer of Ti on the SiC particle, while the EDS analysis revealed the presence of Ti in the outer zone (zone A).

Detailed SEM images of a SiC particle with Ti coating are presented and in Fig. 7. Also EDS analysis were carried out in different zones from the inner to the outer zone (named zones 1 to 4).

The EDS analysis performed in these four points revealed the presence of Si and C coming from the SiC particles. In was observed the presence of Ti, which proves that the SiC particle is covered by continuous Ti PVD coating of a thickness of about 600 nm.

3.3 Morphological and chemical characterization SiC particles with Ti PVD coating and Ni electrocoating

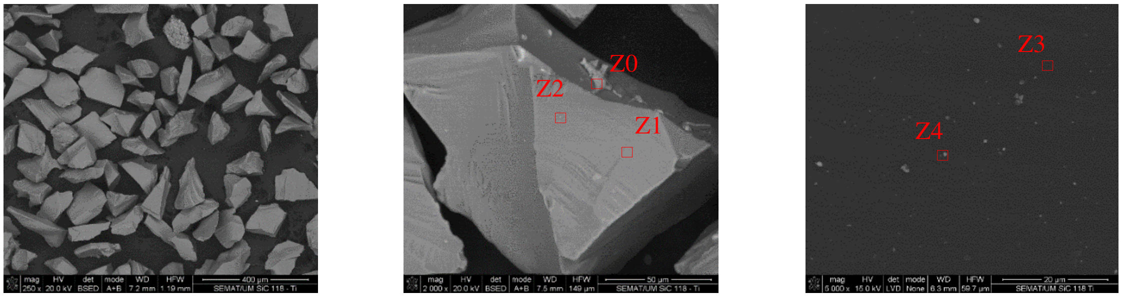
Fig. 8 illustrates the SEM morphology of the SiC particles with Ti PVD coating and Ni electrocoating. EDS analysis was performed in two zones presented in Fig. 8, while the EDS results are presented in Table 5.

Table 5

Chemical composition (wt.%) of the marked zones (Z1 and Z2) in Fig. 6.

	Z1	Z2
C	11.37	28.25
Si	56.08	69.4
Ti	0.41	0.88
O	13.79	1.48
Cl	6.19	0
Ni	9.19	0
S	2.41	0
K	0.56	0

It can be seen from Fig. 8 that there are zones with higher content of Ni coating (Z1), but there are also zones where the particles were Ni was not detected (Z2). It means that there was not a homogeneous coating.



(a) (b) (c)

Fig. 5. SEM images SiC particles with Ti PVD coating.

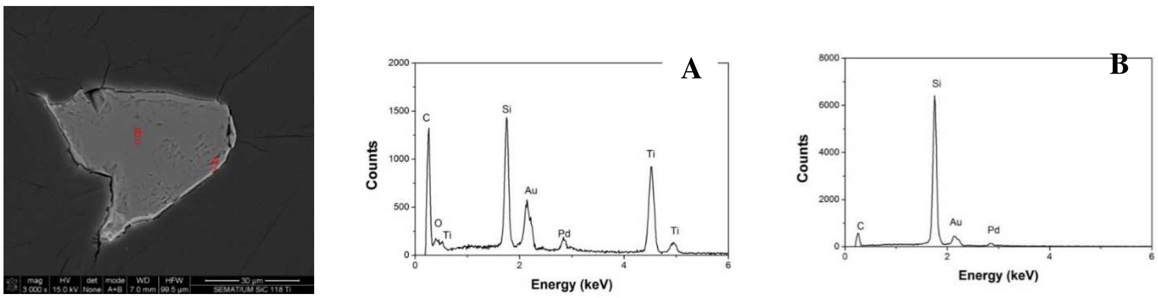
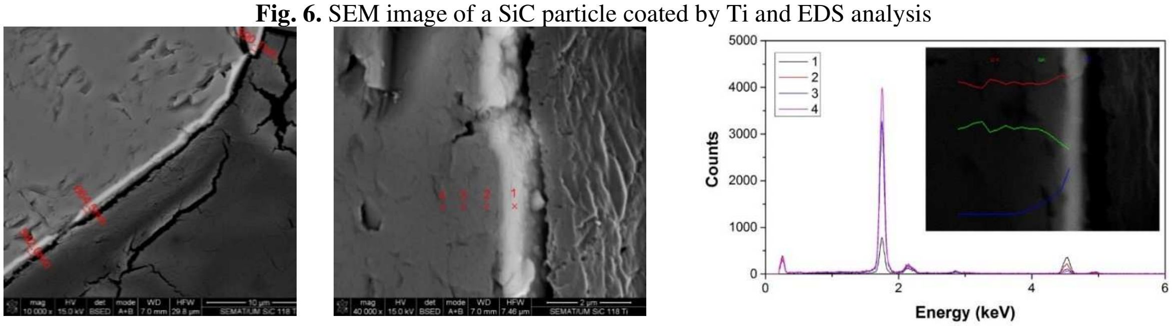


Fig. 6. SEM image of a SiC particle coated by Ti and EDS analysis



(a) (b)

Fig. 7. a. Detailed SEM images of the Ti coating and b. EDS analysis of zones 1 to 4

3.4 Microstructural characterization of the 410L SS/SiC composites

Microstructures of the unreinforced 410L SS and all 410L SS matrix composites are presented in Fig. 9. Fig. 9a shows the typical microstructure of 410L SS. The microstructure indicates that the 410L SS was successfully processed by hot pressing technology.

Fig. 9b shows the microstructure of 410L SS reinforced with SiC particles that were not functionalized. It can be seen that the SiC particle reacted with the 410L SS matrix. There can be observed that there is a completed SiC particles dissolution. This dissolution of SiC particles leads to a transformation of 410L SS matrix martensitic structure transformation into pearlite structure (lamellar). More details regarding the reactivity between the SiC particles and 410L SS are given elsewhere [4].

Fig. 9c shows the microstructure of 410L SS-matrix composite reinforced by Ti coated SiC particles. It can be seen that also in this case there was complete dissolution of the SiC particles. This means that the Ti PVD coating reacted, so it was not effective in avoiding the dissolution of the SiC particles.

Fig. 9d shows the microstructure of 410L SS-matrix composite reinforced by Ti/Ni coated SiC particles. It can be observed the presence of the SiC particles (dark phase), where most of them were not dissolved. This means that the Ni coating was efficient in avoiding the dissolution of the SiC particles.

(a) (b)

Fig. 8. SEM images SiC particles with Ti PVD coating and Ni electrocoating: (a) global view and (b) detailed view of the marked zone C.

3.5 Tribological results

a. Coefficient of friction and specific wear rate

Fig. 10 shows the typical evolution of the coefficient of friction (COF) with sliding distance in the case of unreinforced 410L SS alloy against steel ball.

During the running-in period the COF is increasing very fast till entering in the steady-state regime that will be the same till the end of the test. No significant difference in the steady-state average value coefficient of friction (0.5) existed between all studied cases.

Fig. 11 shows the specific wear rate of 410L SS alloy, 410L SS/SiC composite and 410L SS/SiC-Ti-Ni hybrid composite. The wear performance was strongly influenced by the interface reaction between the coated SiC particles and 410L SS matrix. Wear results showed that the 410L SS-matrix composite reinforced by uncoated SiC particles and 410L SS-matrix composite reinforced by Ti/Ni coated SiC particles presented a smaller wear rate than the 410L SS. In the case of hybrid composite, seems that the use of both Ti and Ni coating on the SiC particle had a beneficial effect, as it exhibited a reduction in the specific wear rate, as compared to all other cases.

b. Surface morphologies

SEM images of the center area of the wear track after wear tests of 410L SS alloy and 410L SS composites samples are shown in Fig. 12. The sliding directions are indicated by arrows in the SEM images.

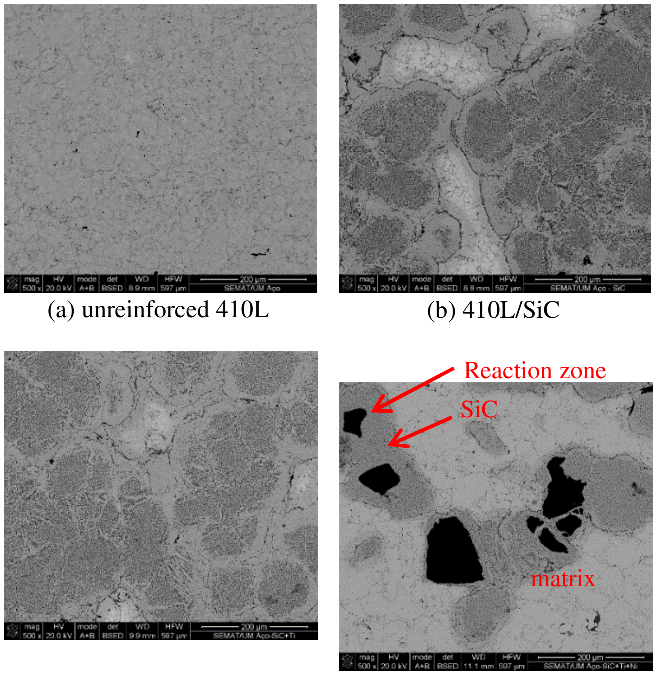
SEM images on the wear track surface (Fig. 12 - magnification 1000×) revealed that there is a higher adhesion in the case of 410L SS alloy (Fig. 12 a) as compared to the composites (Fig. 12 b and c). This may be explained based on the higher chemical affinity between the plate and the steel ball. It can be observed from the worn surface topography of the of 410L SS/SiC composite and 410L SS/SiC-Ti-Ni hybrid composite (Fig12 b and c) that in the region where there was dissolution of the SiC particles no adhesion was observed. In the case of all worn surfaces it can be clearly seen grooves parallel to the reciprocating sliding direction. The grooves seem to be more pronounced in the case 410L SS alloy, followed by 410L SS/SiC composite. Thus, in these cases is present beside the adhesive wear mechanism, also the abrasive wear mechanism.

Regarding the worn surface of 100L SS/SiC-Ti-Ni hybrid composite after sliding against the steel ball (Fig. 12 c), seems smoother in comparison with the worn surface of the 410L SS alloy and 410L SS/SiC composite. The lower level of surface degradation in the case of 410L SS alloy/SiC-Ti-Ni hybrid composite is in accordance also with the specific wear rate value (Fig. 11).

Thus, the lower level of degradation in the case of 410L SS alloy/SiC-Ti-Ni hybrid composite can be explained based on:

(a) *Strengthening effect of the 410L SS matrix by Ti/Ni coated SiC particles* (no pull out mechanism of SiC particles can be observed);

(b) Ti/Ni coated SiC particles act like *load bearing elements* [24]. The Ti/Ni coated SiC particles support the load, so they protect the 410L SS matrix during the reciprocating sliding.



(a) unreinforced 410L (b) 410L/SiC

(c) 410L/SiC-Ti (d) 410L/SiC-Ti-Ni

Fig. 9. SEM images of the composites microstructures.

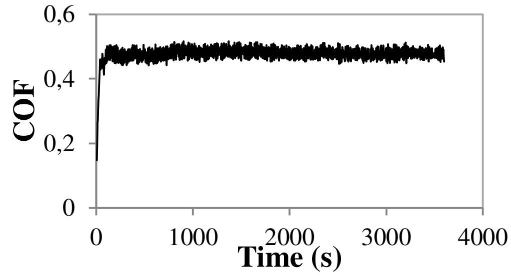


Fig. 10. Evolution of COF along the time for unreiforced 410L SS alloy.

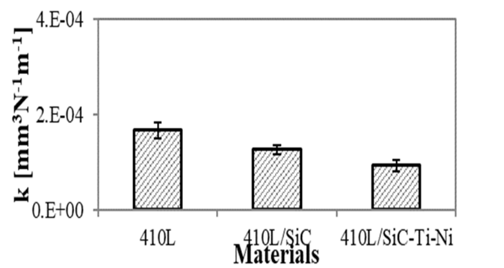
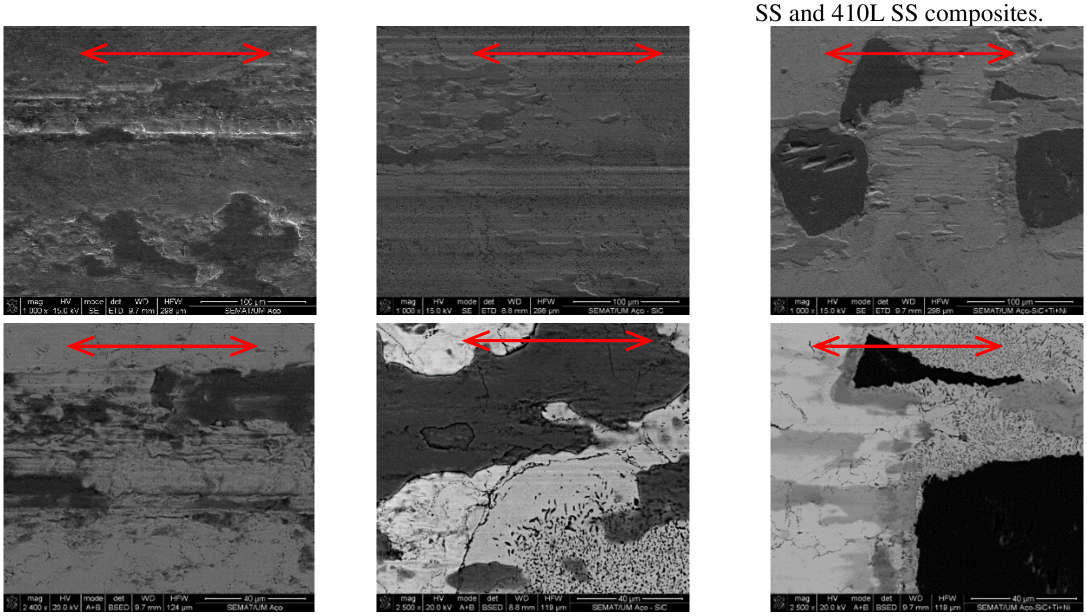


Fig. 11. Specific wear rate for unreinforced 410L SS and 410L SS composites.



(a) 410L SS alloy (b) 410L SS/SiC (c) 410L SS/SiC-Ti-Ni

Fig. 12. SEM images of the center area of the wear track formed after wear tests: 1000× magnification (top line and 2500× magnification (bottom line)

Thus, it can be clearly seen that the combination of PVD coating and electrocoating played an important role also on the production of new composites. In some cases, there is the dissolution of the reinforcement into the matrix, but by controlling the composition of the reinforcement surface it is possible to control the reactivity between matrix and reinforcement, and consequently it is possible to control the mechanical properties of the interface which have a great influence on the global properties of the composites. Also, the use of the PVD allows the production of an electrical conductive coating in nonconductive powders, which allows to use of them in electrocoatings.

4. CONCLUSIONS

From the present investigation on the reactivity of functionalized silicon carbide particles SiCp and 410L stainless steel alloy, the following conclusions can be drawn:

- The Ti PVD coating on SiC particles allowed the Ni electrocoating,
- The Ni coating allowed the reactivity control between SiC particles and stainless steel 410L matrix,
- The 410L SS alloy/SiC-Ti-Ni hybrid composite samples exhibited better wear resistance than the 410L SS alloy and 410L SS/SiC composite samples. This is due to strengthening effect of the 410L SS matrix by Ti/Ni coated SiC particles and to the fact that SiC particles behave like load bearing elements,
- The combination of PVD and electrocoating is a feasible process for reactivity control between reinforcement and matrix, making the production of metal matrix composites more versatile.

5. ACKNOWLEDGEMENTS

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Comportamentul tribologic al compozitelor 410L SS - SiCp armate cu particule SiC functionalizate prin acoperire cu Ti-Ni

Acest studiu se referă la comportamentul tribologic al compozitelor 410L SS - SiCp armate cu particule SiC funcționalizate prin acoperire cu Ti-Ni. Compozitele SS 410L au fost realizate prin tehnica presării la cald. Pentru comparație, aliajul 410L SS și compozitul armat cu SiCp neacoperit - 410L SS / SiC, au fost, de asemenea, testate. Un tribometru bilă-pe-plan cu mişcare alternativă (Bruker-UMT-2) a fost folosit pentru a evalua comportamentul tribologic. Materialele produse au fost testate în contact cu o bilă de oțel. Suprafețele uzate au fost analizate cu SEM/EDS. Rezultatele au indicat faptul că combinația dintre PVD și acoperiri electrochimice este un proces fezabil pentru controlul reactivității dintre armătură și matrice. Rezultatele arată că utilizarea unui strat de Ti-Ni este util în îmbunătățirea rezistenței la uzură a compozitelor SS 410L.

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