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Carbides formation in weld metal produced by recycling of titanium machining swarf

J G Fagundes^{1,3}, V A Ventrella², J Gallego², A M Moreno¹, J L Jácome⁴, and A Q Bracarense¹

¹ Federal University of Minas Gerais, Belo Horizonte, Brazil

² São Paulo State University, Ilha Solteira, Brazil

³ Minas Gerais State University, João Monlevade, Brazil

⁴ Francisco de Paula Santander University, San José de Cúcuta, Colombia

E-mail: gedaelfagundes@gmail.com

Abstract: Application of titanium alloys for orthodontic implants have increase in last years and it has generated a great amount of titanium chips which are disposed as metal scrap. Titanium carbide has high hardness and can be applied as coating reinforcement against wear. Chips of titanium alloys were processed and used in the fabrication of experimental hardfacing consumables for improving the resistance of wear of mechanical components. Coatings were produced in cast steel specimens using the gas tungsten arc welding process. Microstructure was investigated by means of scanning electron microscopy. Microhardness of the coatings was analyzed by a dynamic ultra-micro hardness tester. The microstructure of the welding metal was composed of circular particles of titanium carbide surrounded by a ferritic matrix. The measures made by electron dispersive spectroscopy showed a variation in titanium amount trough the weld, where larger contents of titanium were found, and the matrix. The crystallographic analyses reinforced the formation of titanium carbides embedded in a ferrite matrix into welding metal.

1. Introduction

Metallic carbides have been applied as a prevention of damaged parts caused by wear phenomenon. It is reported by literature, wear is the manly responsible for losses and failure in equipment and parts at the mechanical industries. According some studies the economic losses due to wear, friction and corrosion is about 4.5% of the gross national product (GNP) [1]. Other reports have indicated wear as a responsible for almost 50% of failure occurrence where-as abrasive is the wear mechanism more frequently and most aggressive [2]. Aiming to prevent wear injury by abrasive mechanism, surface parts are reinforced by techniques of hardfacing by addition metal compound of hard particles or elements which will reacts into hard particles during welding pool formation, resulting in a weld formed by a matrix embedded with a hard second phase such as carbide. Carbides has high hardness, chemical stability and high melting point [3].

Commercially, chromium is the most used element (ternary systems Fe-C-Cr) inside an addition metal welding to reacts in carbide form, Cr₇C₃ (2150 HV). It is attributed by its lower cost into fabrication processes when compared to other elements which react as a carbide and could be applied for wear requests [4]. Although, various studies have demonstrated that other kinds of carbides have shown high hardness and better wear resistance when compared to chromium carbides, such as tungsten



(2350 HV), vanadium (2900 HV), titanium (2800 HV – 3200 HV) [3,4]. A layer composite applied on a surface owns a high hardness on a low carbon or stainless-steel substrate with lower mechanical strength or high wear rate [4,5]. Hardness is considered the most important mechanical property of wearing material; however it acts as an indicative of wear resistance. Holleck has attributed abrasion resistance also as a result due to the ability of the material to reduce internal stresses [4].

In other hands, some titanium alloys have been designated for orthodontic implants and surgical prosthesis. ASTM F67 (pure titanium) and ASTM F136 (Ti6Al4V) alloys have been widely used for these parts on to machining processes fabrication [6,7]. As a machining process results, it has been generated a wide amount of chips. There is some estimated consideration about 40% (volume) of material are converted into chips. Some implants companies discard each one, about 5 ton of titanium chips alloys per year. Chips has lower cost and are discarded as scraps. Alloys described above own at least 90% of titanium (nominal chemical composition) and the chips generated deserves some attention for the fact that these materials could be insert at the production cycle again as a raw material for welding consumables aiming to form titanium carbides, improving the service life of parts susceptible to wear mechanisms.

The present study aims to investigate the application of titanium chip as a flux compound added with graphite which will be layered on to a substrate forming a previous layer moisture and then melted by a GTAW process. Therefore, titanium chips are going to be inserted as a raw material for welding fluxes attributing a sustainable and economic alternative for these residues.

2. Methodology

The fine of titanium were obtained by milling chips proceeding from machining process of ASTM F67 and ASTM F136 alloys. Initially the chips were cleaned in a water and detergent solution, Table 1, for removal impurities as cutting fluid, proceeding from machining. Chips were kept under immersion solution for at least 24 hours. After that the chips were purified in an ultrasonic cleaner to promote a more efficiently cleanness. Sequentially the chips were kept in a dryer.

Table 1. Steps of cleanness chips.

Step	H ₂ O (l)	Detergent (ml)	Time (min)
1° Immersion	10.0	400	1440
2° Immersion	10.0	400	1440
1° Ultras. Cleaner	0.5	20	5
2° Ultras. Cleaner	0.5	-	3

Chips were ground by an experimental apparatus built to be fixed in a column drill machine, Figure 1. This apparatus was a gradual development when compared to the previous device made of only metallic pieces [8]. Its principle of work is made by the rotating movement of the handle while the chips are under pressure into the milled chamber, between the granite pieces. Fines separation and control of particles size were made by a grid whereas particles less than 40 Mesh has become a compound used as fluxes.

The ratio of titanium chips and graphite powders were combined by the molar ratio of TiC (molTi:molC = 1:1). According stoichiometry, the proportion Ti:C by the weight is about 10:2.5. However, intending to ensure that complete titanium reacts as carbide and expect occasional losses, final weight ratio of Ti:C was defined 10:3. Blended powders were mixed with a 2% sodium silicate. Table 2 shown the nominal chemical composition of flux which was pre-placed on the surface of substrate by using a mold to form a layer, Figure 2. Dimensions of layer are 2.5 mm x 6 mm x 76.2 mm. A low carbon bar was used for the substrate material. The dimensions of substrate are 10 mm x 28 mm x 76.2 mm. After pre-placed, substrate with layer were kept in a dryer to eliminate water present in the flux.

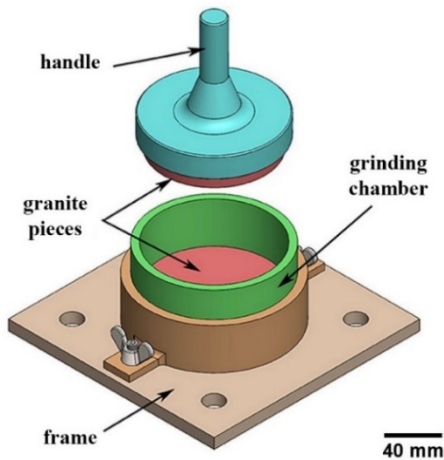


Figure 1. Apparatus developed to grind titanium chips.

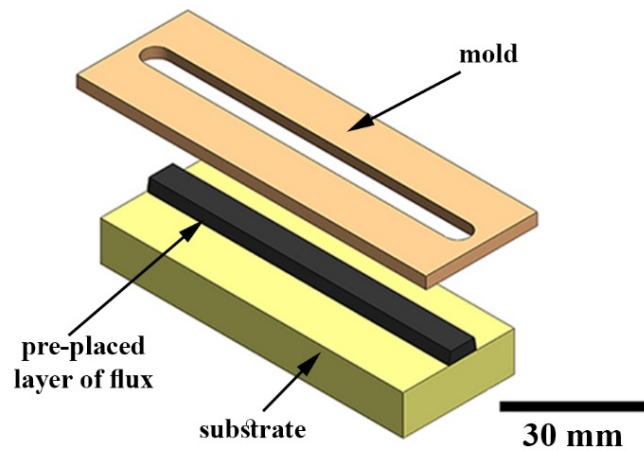


Figure 2. Pre-placed layer of mixture with Ti and C.

Table 2. Chemical composition nominal of flux used as a pre-placed layer (weight).

Alloy	Ti	C	Al	V	Others
ASTM F67	Bal.	22.6	0.0	0.0	2.5
ASTM F136	Bal.	22.6	4.5	3.0	2.4

Welding was conducted by using a GTAW power source of Lincoln Electric, model precision TIG 225. A portable car of White Martins, model MC46 was used to conduct the GTAW torch, keeping the welding speed under control. Welding parameters were determined by experimental results. After some preliminary tests, welding parameters were defined as shown in Table 3. For preventing unstable effects at the moment of open and close electrical arc, a low carbon bar of 12.7 mm was positioned at both extremity of substrate to avoid any kind of disturbance on weld, Figure 3.

After welding, samples were cutting from the alloyed specimens for macrography examination, microstructure characterization and hardness measurement. Samples were identified as P1, P2, P3, P4, P5 for the layer melted using an ASTM F67 alloy chips and L1, L2, L3, L4, L5 for the layer melted using an ASTM F136 alloy chips, Figure 4. Samples P5 and L5 were prepared for X-ray diffraction.

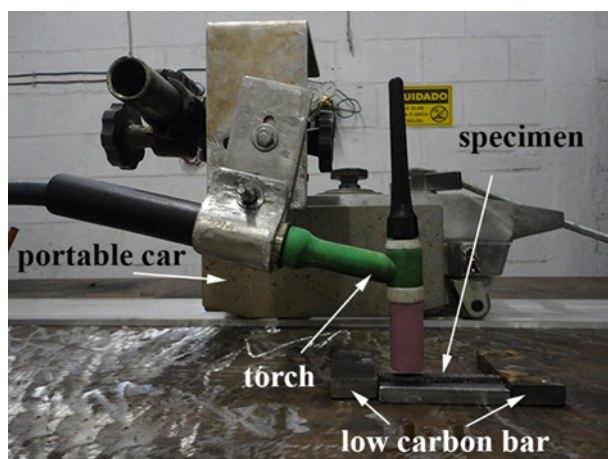


Figure 3. Photo of positioned torch and low carbon bar at the substrate extremity.

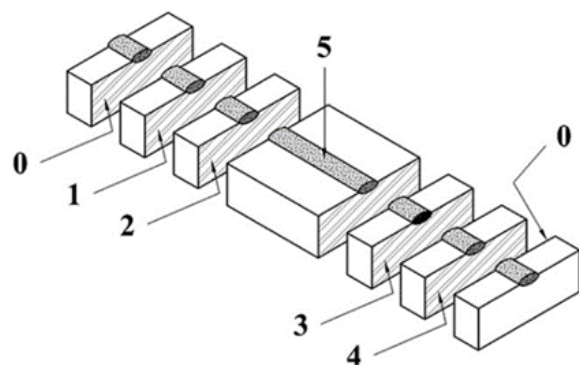


Figure 4. Indication of the sections of specimens in weld bead.

Table 3. Welding parameters.

Current (A)	215 – 230
Welding Speed (mm/min)	100 ± 3
Flow Argon (l/min)	12 – 15
Tungsten electrode	W-Th 2%
Polarity	DCSP

Samples were imbedded in bakelite and prepared by conventional means of metallographic examination. Nital 2% was used to etch the surface of samples.

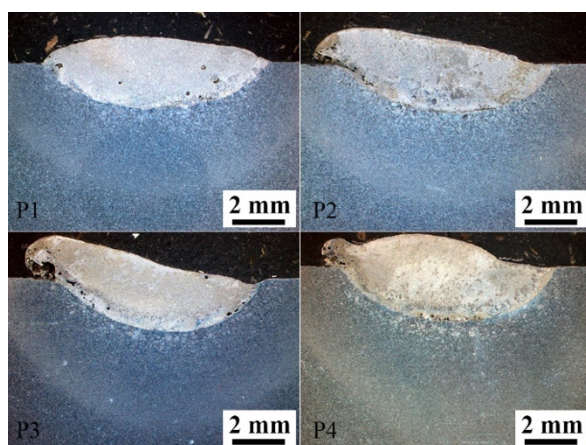
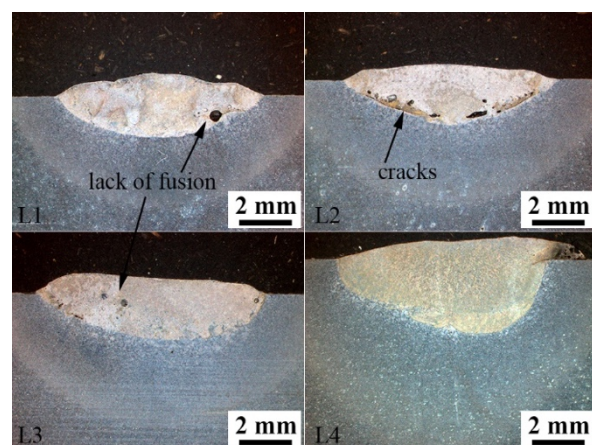
Macrography was taken by a ZEISS DISCOVERY V8 stereomicroscopy. By these images, weld bead geometry, heated affected zone and dilution ratio, which determine the substrate proportion that move to the weld pool during melting point, were evaluated.

Microstructure characterization was conducted by a conventional scanning electron microscope by means of energy dispersive spectroscopy (EDS) and backscattered electrons (BSE). Phases of the weld were evaluated in a SIEMENS DL-5005 diffractometer with a $\text{CuK}\alpha$ radiation and graphite monochromator. Diffracted intensity for the specimens has been registered at the range of 20° to 100° by the scanning speed of 2 degrees/minute. Crystallographic files information (CIF) from Inorganic Structural Database Information were employed to identify the diffractogram generated by the specimens.

Micro-hardness was measured by using a SHIMADZU DUH 211S whereas the hardness was determined by equivalent indentation according ISO 14577 [9]. Equivalent indentation HV^* is evaluated by the maximum load considering the Vickers indentator as an ideal geometry. Load of 1 N ($\text{HV}0.1$) kept for 5 seconds was used to measure hardness starting from the top of weld bead to the substrate above the heat affected zone. For statistical analyses, the value of hardness was taken from four measurements.

3. Results and Discussion

Macrography of the section samples (P1 – P4 and L1 – L4) are shown in Figure 5 and Figure 6. It can be observed some discontinuities inside de weld like as porosities, lack of fusion and cracks at the interface of weld and substrate. Porosities and lack of fusion could be consequence of the welding speed combined with solidification rate and some impurities (oxides) present in the flux. Vapor generated during the welding pool formation does not have enough time to escape from the liquid metal. Cracks can be nucleated and propagated as a result of high welding current (230 A) associated with high solidification rates [10]. Experimental tests to form welds with welding current lower than 230 A did not form any adequate samples to analyses.

**Figure 5.** Samples P1-P4 (ASTM F67).**Figure 6.** Samples L1-L4 (ASTM F136).

Current is the most significant welding parameter which can affect the geometry of weld bead, penetration and dilution rate [11]. Dilution rate in the samples obtained values above 55%. According literature dilution for coating welded by GTAW must reach values at the range of 10% to 20% [12]. It also can be verified an increasing of dilution throughout the weld as a result of lower heat dissipation which acts raising heat input during welding. Elevated dilution decreases the amount of addition metal (flux) in the weld exposing a more effects of substrate properties into the coating resulting in a low presence of carbides decreasing the hardness [13].

Figure 7 and Figure 8 shows the image of microstructure generated by secondary electrons of the section sample in central regions of both samples, according design showed in Figure 6. It can be noted a precipitate of TiC as a second phase embedded in a ferrite matrix. Areas at the center of the weld show more coarse TiC particles as the same reported by Lima *et al.* studying weld formed by chromium carbide [14]. During the weld solidification, regions near to the fusion line show high solidification rates. Then, reactions of carbides are interrupted when temperature reaches values which carbide formation are not favorable. Although, at the central regions of the weld, the solidification rate of the liquid is slower than others. Reaction among titanium and carbon acts for more time and it promotes the coarse carbide formation.

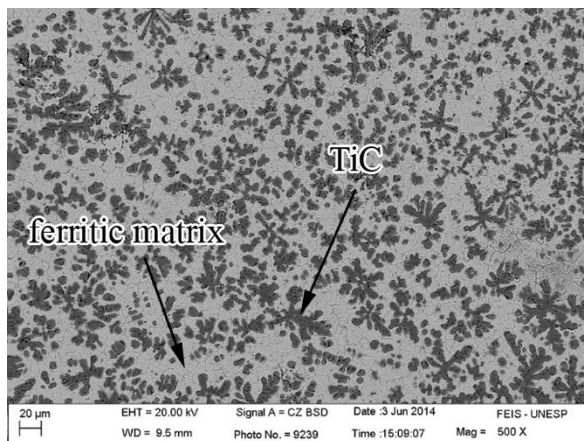


Figure 7. Presence of TiC in microstructure of weld by using ASTM F67 titanium chips.

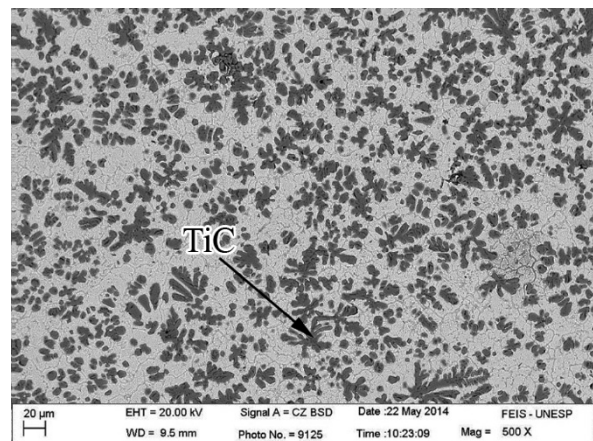


Figure 8. Presence of TiC in microstructure of weld by using ASTM F136 titanium chips.

Both titanium chips alloys applied as raw material (ASTM F67 and ASTM F136) for the flux, reacted as the form of carbide. Fraction area of carbides were determined by free software (ImageJ). Areas less than $5 \mu\text{m}^2$ were dismissed. Fraction area of carbides were calculated through the images the micrograph and the average size for both alloys used was $37.2\% \pm 3\%$. Regions close to fusion line reached lower value. It can be attributed by the substrate dilution acts more significantly effects into weld. Fraction area at the fusion line obtained values of $26.7\% \pm 7\%$. These data were very similar to the study reported by Xinhog *et al.* using ferrotitanium element as flux which exposed values at the range of 25.4% to 31.6% [15].

Chemical composition of carbides was evaluated for EDS. Precipitates of second phases is compound of titanium and carbon majority. Sample of ASTM F136 has identified vanadium inside the carbide. Holleck reported that the binary carbide of the transition metals is in most cases completely miscible in solid states and often show extreme properties for ternary or quaternary compositions [4]. It is the purpose of vanadium are localized inside the particles reacting as solid solution with TiC.

Microhardness of the weld was measured along the depth from the top in a step of 0.2 mm, Figure 9 and Figure 10. All the data were an average of four measurement take of each sample. Presence of TiC results in a notable increasing of the hardness at the surface of substrate. Hardness of weld deposited with chips of ASTM F67 (pure titanium) showed more increased in values when compared to ASTM F136 which is attributed due to the more offer of titanium. Both welds gradually decreased

It can be seen that the micro-hardness of the weld gradually decreased with increase of distance from top of the weld bead. This may be attributed to the different-density-driven force. Compared with the Fe-based alloy, the relative lower density of TiC particles ($4.90 \text{ g/cm}^3 - 4.93 \text{ g/cm}^3$) tended to segregate to the upper regions in the weld. The gradient distribution of TiC particles led to a gradual hardness distribution of the coating resulting in high hardness at the regions where fraction of TiC is more evident. As a consequence of a ferritic matrix (low hardness) mixed of TiC (high hardness) as a second phase, deviation at the hardness measurement is a result of the region reached by the Vickers indentator. Regions with more fraction of TiC results in high resistance to indentator penetration.

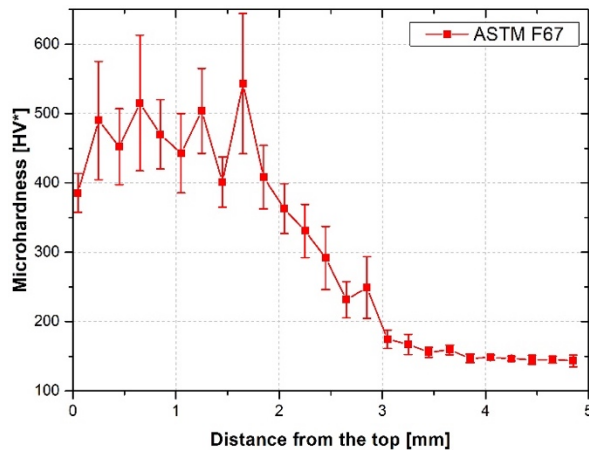


Figure 9. Microhardness of the weld deposited by ASTM F67.

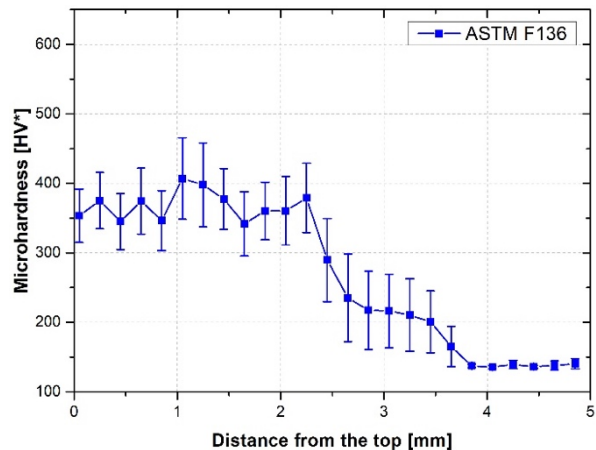


Figure 10. Microhardness of the weld deposited by ASTM F136.

4. Conclusions

Application of titanium chips as a raw material aiming to form TiC into weld deposited on the surface of substrate and melted by GTAW processes has been showed as a possible and attractive means to form a coating to prevent losses by wear due to microstructure revealed. The proposal methods of applying a pre-placed layer has been formed a weld with dilutions rates highest than those levels recommended by literature. Although weld has reached high fraction areas of TiC and consequently elevated hardness values were found due to the elevated offer of titanium proceeding from the chips, mainly those came from pure titanium alloys. These results are expected to provide an enhanced at the properties of wear resistance. Pores, cracks and lack of fusion also have been found in the weld.

Acknowledgments

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