



Graded metal-carbide composites produced by solid state transformations in VC-coated Ni

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ABSTRACT

Graded metal-carbide composites were produced by solid state transformations in Ni samples, which were coated with VC by thermally activated chemical vapor deposition (CVD) onto a Ni substrate, followed by heat treatments. CVD produced a multi layer coating, an inner M₂C layer and an outer V–Ni–C solid solution, with randomly dispersed M₂C particles. Also, a V enriched Ni substrate converted to Ni₂V and/or Ni₃V intermetallics, up to a depth of about 15 μm. Further heat treatments reduced the amount of intermetallics, and precipitated M₈C₇ that increased with time. These phase transformations were very intense above the order–disorder transformation temperature for the Ni₃V intermetallic (1045 °C), and drastically changed the microstructure, producing a cermet layer, i.e., M₈C₇ precipitated particles in a Ni matrix. The observed microstructural modifications suggest the possibility of tailoring a graded material for wear control, conformed by an external thin carbide layer, followed by a thicker cermet zone.

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1. Introduction

Vanadium carbide (VC) has a reportedly outstanding tribological behavior, considering the combination of a high abrasion resistance with a low-friction coefficient. VC abrasion resistance is directly related to its high hardness, which is superior to all other similar compounds of transition metals in groups IV B and V B, except TiC [1]. Beside, VC has a fast oxidation kinetics that leads to the formation of vanadium oxide and oxycarbide surface layers, which are characterized by low shear strength and friction coefficient, comparable to the ones of solid lubricants [2–4].

Despite the attractive tribological properties, industrial applications of VC are still limited. It is included as a minor constituent in cemented carbides, to enhance wear performance, to inhibit grain growth and increase density [5–10]. The best known application of VC is the coating produced by vanadizing steels in molten salts bath, called *Toyota process*, which was proposed in the early '1980's [11]. The carbide layer grows through reaction between vanadium atoms, supplied for the bath, and carbon from the substrate. To be applied, the process requires a substrate containing an important amount of carbon, usually high carbon steel [11,12].

There are other processes widely used for hard coatings production, such as PVD and CVD, which might avoid substrate depletion

in carbon or allow the use of carbon free substrates, CVD being more attractive when uniform coatings are required on surfaces with complicated geometries, such as forming or casting dies. Nevertheless, they were scarcely explored for VC deposition.

Interesting surface engineering options, consisting in the in situ formation of relatively thick layers of graded steel-VC composites, were more recently advanced. A new cast sintering process was proposed by Wang et al., where the solidification of steel substrate is combined with surface sintering of ferrovanadium and carbon, forming carbide reinforcements [13,14]. Another recently proposed process was laser assisted VC incrustation in steel substrate, which produced a well adhered composite coating, crack and porosity free; this graded composite is promoted for enhancing service life of die-casting dies [15].

It is worth to note that tailoring graded VC composites coatings on metal substrates (M) requires the understanding of solid state transformations in the V–M–C system, which is better dominated in the case of steel substrate, i.e., V–Fe–C system [11–17]. For other V–M–C systems, little work has been done. Regarding Ni–V–C system, a detailed crystallographic characterization was provided by Viswanadham and Precht; they stated the possibility of Ni being contained in well defined VC_x structures (where x takes the values: 0.75, 0.83 or 0.87), as well as the multi-component character of the binder, which contain Ni–V–C solid solution and intermetallic compounds [18].

The present work proposes a different route to produce graded composites containing VC, namely thermally activated CVD

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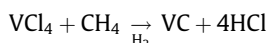
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followed by heat treatments. The composite layers are produced on a Ni substrate, whilst Ni–V alloy constitutes the composite matrix. The solid state behavior of the V–Ni–C system is also approached and related with the possibility of in situ forming VC–(Ni–V) composites.

2. Experimental

For the preparation of the coated samples, 25 mm × 25 mm × 6.35 mm slices of 99.99% Ni, were employed as a substrate. Previous to CVD, the surface to be coated was polished on SiC paper to 1000 grit and ultrasonically cleaned in perchlorethylene and isopropanol. The CVD process here employed was a variant of the classical thermal activated CVD for TiC deposition from chloride precursors, which was developed and commercialized by CSEM in the '1970's [19,20]. To deposit VC, an evaporator for VCl₄ precursor has been built and adapted on a CSEM industrial type CVD reactor. Process precursors were VCl₄ and the gases: 99.995 vol.% CH₄ and 99.95 vol.% H₂, both with water contents lower than 5 ppm. Besides, 99.998 vol.% Ar, with less than 3 ppm water content, was used to purge and fill the CVD reactor. The overall chemical reaction, governing the process, was assumed to be:



at 1000 °C and 7 MPa. To obtain VC coatings with a nominal thickness of 5 μm, the deposition time was 3 h. Further cooling was performed inside the CVD reactor, in an argon atmosphere.

The coated samples were heat treated at 850 °C and 1000 °C for 45 min and at 1150 °C for 5, 30, 45, 60, 120, 180 and 720 min. Heat treatments were performed in argon atmosphere, with heating and cooling rates of 10 °C/min and 8 °C/min., respectively. Cross sections of the samples, coated and heat treated, were metallographically prepared by polishing on SiC paper to 1200 grit, then to 1 μm with diamond suspension and, finally, to 0.05 μm with colloidal silica.

To identify phases, conventional XRD was performed on a Philips general-purpose system, by using a Cu anode, at an operating power of 40 kV and 20 mA. For the micro structural characterization, a JEOL 7810 equipment was used, which combines secondary electron (SE) and back scattered electron modes (BSE) for the morphological observation with Auger electron spectroscopy (AES) for the evaluation of the surface chemistry. Punctual chemical analysis spectra were produced by an Auger electron micro-probe, with a spot size less than 1 μm. At least three punctual analyses were performed in each phase. Previous to the analysis, an Ar ion beam, 3 keV accelerated, eroded the sample surface at a rate of about 50 nm/min. The main purpose of this procedure was to clean the surface, especially to remove any possible oxygen contamination. The Auger electron spectra were employed to quantify element contents, considering one reference peak per each element, with the following characteristic energies (in keV): 271 for C, 470 for V, 500 for O and 845 for Ni. Maximum intensity values of the reference peaks were found in the spectra of reference samples of C (graphite), Ni, V, SiO₂, and WC, which were analyzed under the same conditions with the ones used during the evaluation of the coated samples.

3. Results and discussion

3.1. The as-coated samples

The CVD process here employed produced a rather complex system. Optical and scanning Auger microscopy (Fig. 1), along with chemical analysis by AES (Table 1), showed that the system was a double-layer deposit of about 5 μm in thickness, corresponding

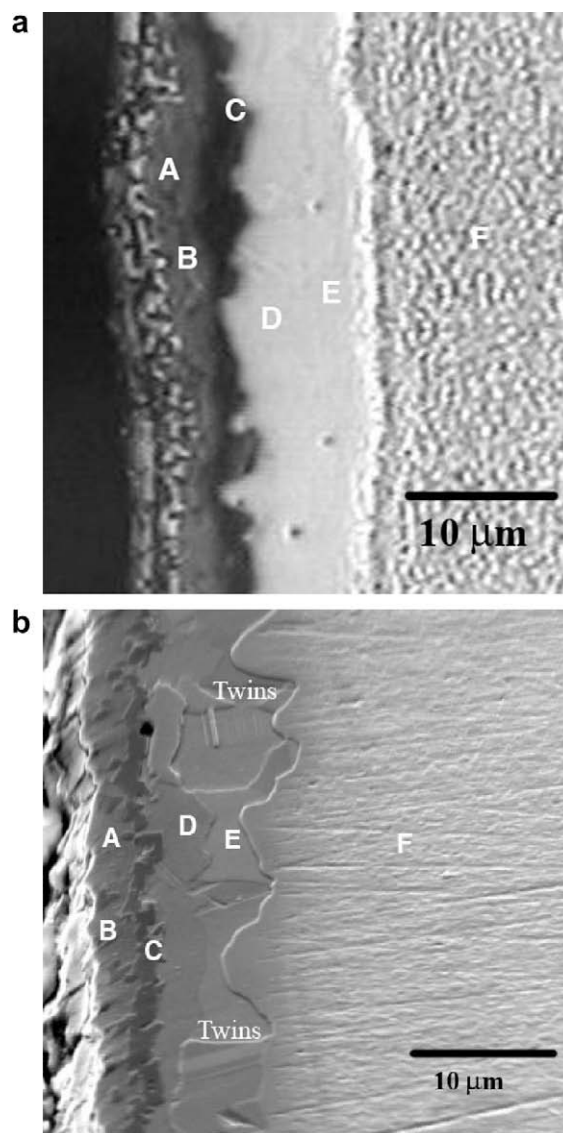


Fig. 1. Cross section of the as-coated sample, showing the points where chemical analysis were performed.

Table 1

Chemical composition (at.%) in the phases which were identified in Fig. 1. AES punctual analysis.

Regions and phases	C	V	Ni	O	Ni/V
A: External layer matrix (assumedly V–Ni–C solution)	15	53	25	6	0.5
B: Discreet carbides in the matrix (assumedly M ₂ C)	26	57	13	3	0.2
C: Continuous carbide layer (assumedly M ₂ C)	25	50	21	4	0.4
D: Assumed to be Ni ₂ V intermetallic	0	35	62	2	1.8
E: Assumed to be Ni ₃ V intermetallic	6	21	69	4	3.3
F: The Ni matrix	0	0	87.5	2.5	–

to the CVD coating, and a modified-substrate layer, around 10 μm thick.

The upper region of the double-layer deposit was 3–4 μm thick, and composed by randomly dispersed sub-micron carbide particles, immersed in a V–Ni–C matrix (Regions A and B in Fig. 1). The chemical composition of the dispersed particles was in the range of the M₂C phase (M = V + Ni). The bottom layer, in the interface between the CVD coating and the Ni substrate (Region C in Fig. 1), was about 1 μm in thickness, conformed by an almost

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