

# ANALYSIS OF DIFFERENT SURFACE PREPARATION PROCEDURES FOR DEPOSITION OF POLYCRYSTALLINE DIAMOND COATINGS ON CEMENTED CARBIDES

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**Abstract:** *Cemented carbide has several interesting applications as tools or even as mechanical parts in the mechanical industry, e.g. metal forming, machining as well as mining tools. Among others compositions, tungsten carbide (WC) with 6% cobalt as binder phase, can be regarded as a composition with a suitable balance of toughness and tribological properties (abrasion and adhesion resistance). An improvement on the tribological properties can be achieved by coating of a thin diamond film on top of the structure that makes the contact with the treated material, extends the tools lifetime dramatically. A special concern here is the cobalt content, or even the chemical composition of the binder phase at the substrate that induces bad adhesion of the diamond film. Hence special treatments are necessary to remove the cobalt from the surface – and also immediately below it. In this work, several pre-treatments have been investigated and it is shown that a combination of immersion in a 10 g K<sub>3</sub> [Fe (CN)<sub>6</sub>] + 10 g KOH + 100 ml H<sub>2</sub>O and then in a 6 H<sub>2</sub>O + 3 HCl + 1 H<sub>2</sub>O<sub>2</sub> mixture results in excellent adhesion of the polycrystalline diamond film that was deposited afterwards.*

**Key-words:** *Hard coatings, diamond, hardmetals, surface coatings.*

## 1. INTRODUCTION

The increasing demands on the performance for tool materials used in machine design and manufacturing processes, as well as some aspects as economic reasons, unique properties or engineering and design suitability are supporting new development on the surface processing techniques, especially the coatings technology for the surface or near surface regions of classical materials used in tools and mechanical parts. A typical tool material with broad field of use, including tools for metal forming, machining as well as wear parts, are the cemented carbides, characterized as composite materials, composed by a hard phase (typically, a WC an hexagonal carbide, the phase responsible for the hardness and abrasion resistance) “cemented” by the a binder phase (typically, a metal from the iron group, predominantly cobalt). This composition is processed using a powder metallurgical route, by a liquid phase sintering technique, aiming a microstructure without sub-stoichiometric carbides (eta phase or W<sub>2</sub>C), free carbon, carbide grain coarsening or formation of cobalt pools on a non smooth distribution of the micro structural phases (carbide and binder phases) [1]. Since the introduction of chemical vapor deposition in the seventies, the coating treatments for cemented carbides are experiencing a great progress, and the good results of coatings as TiN, TiC and multi-graded ones, triggered the possibility of polycrystalline diamond coatings on the surface of cemented carbides as an important research topic, with great industrial importance, demanding studies on the process development (reactors and control engineering) as well as

material and surface interface aspects, where the surface preparation and activation processes play an important role. This research topic has been since the 90<sup>s</sup> object of previous works of the authors [2-5] as well from others researchers [6-38]. Some preliminary results as well as a more detailed discussion can be seen at [39-40].

## 2. EXPERIMENTAL

The cemented carbide samples were 5 mm diameter cylinders, composition 94% tungsten carbide (WC) and 6% cobalt. They were cut from a bar by diamond disk and had a height of 3.5 mm or 5 mm. The equipment used for the diamond deposition was microwave plasma assisted chemical vapor deposition reactor [Salvadori et alii 1995] Microwave power, generated by a magnetron, enters a cylindrical resonant cavity from the top. This power activates the reactant gases, which are at a low pressure inside a quartz Bell jar. The cavity base plate has a window through which the rear of the hot surface can be viewed, allowing pyrometric temperature measurements. A moveable sample holder allows control of the sample position in relation to the plasma ball. The deposition conditions were always the same: hydrogen flow: 300 sccm, methane flow: 3 sccm, substrate temperature: 840 °C, chamber pressure: 67 Torr, deposition time: 8 hours. The Scanning Electron Microscope used for the characterizations was a JEOL JSM 6460 LV, with micro analyzers. Samples were never coated with gold.

## 3. RESULTS AND DISCUSSION

Figure 1 shows the EDX spectrum after cleaning of the hard metal samples by immersion in acetone in ultra-sound during 10 minutes. One may clearly observe the tungsten (W), carbon (C) and cobalt (Co) peaks. The different etching sequences were applied. Firstly a preparation step was performed in order to open up the tungsten carbide particle contact net (contiguity) in order to enhance subsequent etching of the cobalt. This step consisted of immersing the samples in a KOH ultra-sound bath or in a mixture of 10 g  $K_3 [Fe (CN)_6]$  + 10 g KOH + 100 ml  $H_2O$ , also in an ultra-sound bath, always for 20 minutes. Figure 2 shows the EDX spectrum after etching with the 10 g  $K_3 [Fe (CN)_6]$  + 10 g KOH + 100 ml  $H_2O$  mixture.

This spectrum shows the same peaks, and also an oxygen peak, indicating oxidation of the sample. The cobalt peaks, however, are much higher than before the etching. This indicates that the etching opened the tungsten carbide structure and that the cobalt is now more exposed. It is certainly not possible that there is now more cobalt in the sample than before the etching! After KOH etching, the EDX spectrum was similar, also including the oxygen peak, but the cobalt peaks were much less pronounced.

Then the prepared samples were etched in three different inorganic acid mixtures : 6  $H_2O$  + 3 HCl + 1  $H_2O_2$ , 1  $HNO_3$  + 5  $H_2O$ , 3 ml  $H_2SO_4$  + 88 ml  $H_2O_2$ , all during 10 minutes, in an ultra-sound bath. Figure 3 shows an EDX spectrum for the sample etched with the HCl based solution, after preparation with the 10 g  $K_3 [Fe (CN)_6]$  + 10 g KOH + 100 ml  $H_2O$  mixture. No cobalt peaks can be found anymore and also the oxygen peak has disappeared.

The samples were also analysed by electron microscopy. And this analysis showed that the microstructure of the samples depended very much on the different etching solutions.

Figure 4 shows the surface of the hard metal after 20 minutes of treatment in pure KOH, in an ultra-sound bath. The surface has been affected very somewhat, some stains and etch pits can be seen; it is still possible to see the grooves made during the polishing of the hard metal surface.

Figure 5 shows the surface of a hard metal sample after 20 minutes treatment in a 10 g  $K_3 [Fe (CN)_6]$  + 10 g KOH + 100 ml  $H_2O$  mixture. One may observe much more etch pits, more stains, the surface looks rougher comparing with the KOH treated sample. Clearly, this process was more aggressive and modified the surface more than the KOH treatment did. Most SEM analysis efforts were therefore concentrated on the samples treated by the 10 g  $K_3 [Fe (CN)_6]$  + 10 g KOH + 100 ml  $H_2O$  mixture.

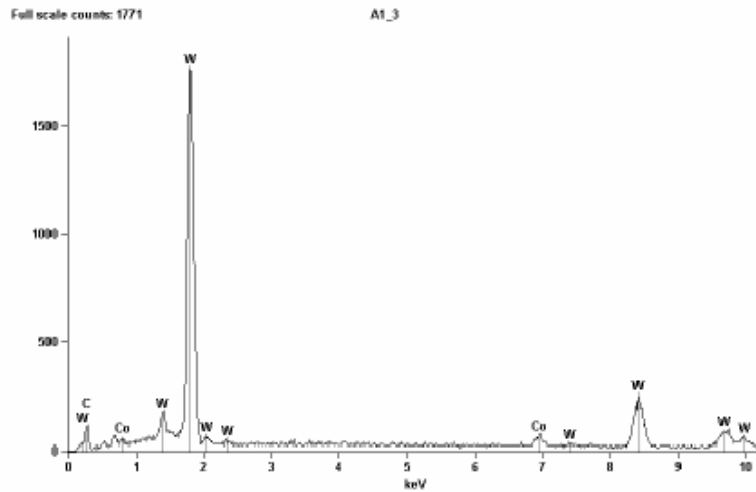


Figure 1: EDX spectrum after cleaning in acetone.

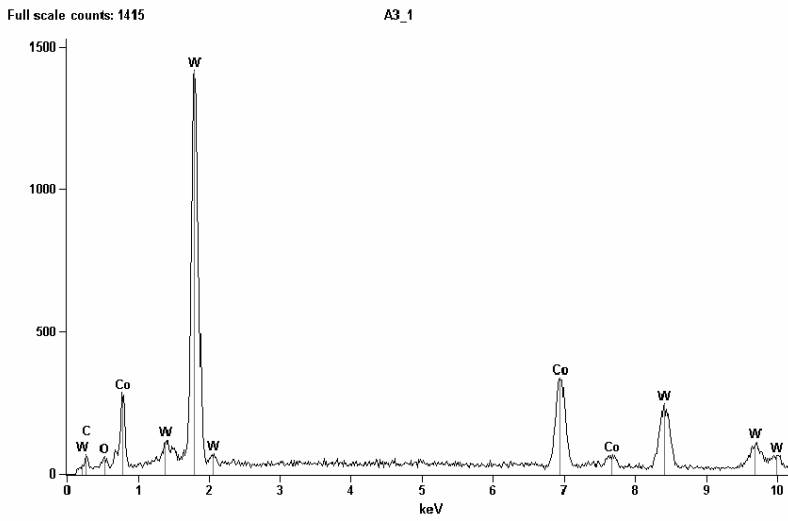


Figure 2 : EDX spectrum of hard metal sample after etching in 10 g K3 [Fe (CN)6] + 10 g KOH + 100 ml H2O mixture.

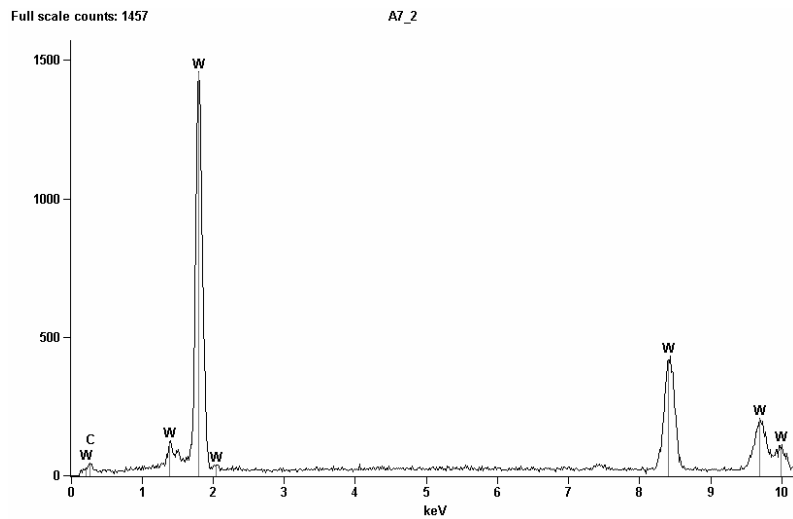


Figure 3 : EDX spectrum of hard metal sample immersed in a 10 g K3 [Fe (CN)6] + 10 g KOH + 100 ml H<sub>2</sub>O and then in a 6 H<sub>2</sub>O + 3 HCl + 1 H<sub>2</sub>O<sub>2</sub> mixture.

The EDX spectra of all the samples etched in (one of ) the three acid solutions looked similar, no cobalt peaks can be found anymore. This indicates that the three investigated mixtures etch cobalt.

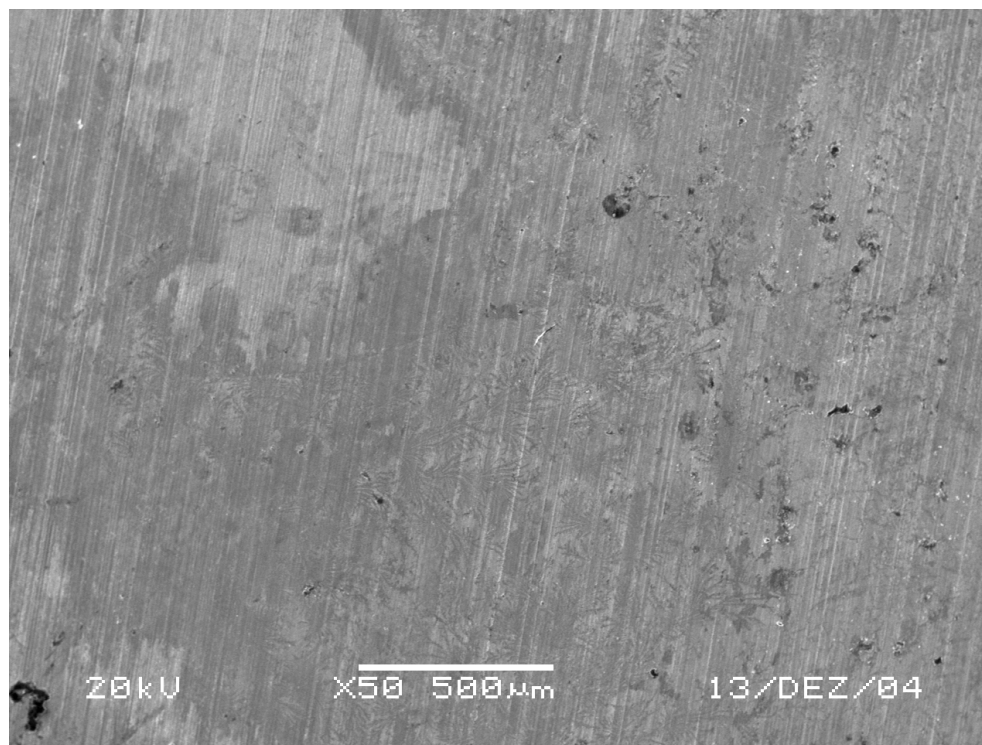


Figure 4 : Surface of hard metal sample after 20 minutes treatment in pure KOH.

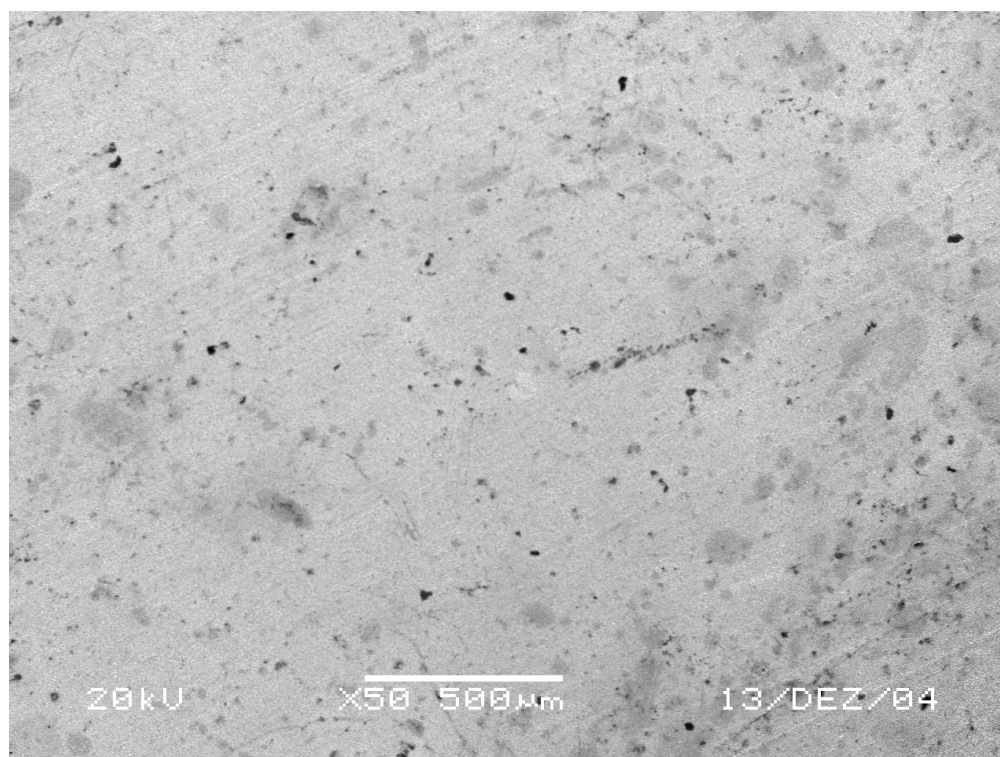


Figure 5 : Surface of hard metal sample after 20 minutes treatment in a 10 g K<sub>3</sub> [Fe (CN)<sub>6</sub>] + 10 g KOH + 100 ml H<sub>2</sub>O mixture.

Figure 6 shows the surface after immersion of the sample in a 10 g  $K_3 [Fe (CN)_6]$  + 10 g KOH + 100 ml  $H_2O$  and then in a 6  $H_2O$  + 3 HCl + 1  $H_2O_2$  mixture. One may clearly observe that the surface has been strongly affected by the etching processes, resulting a porous, spongy structure.

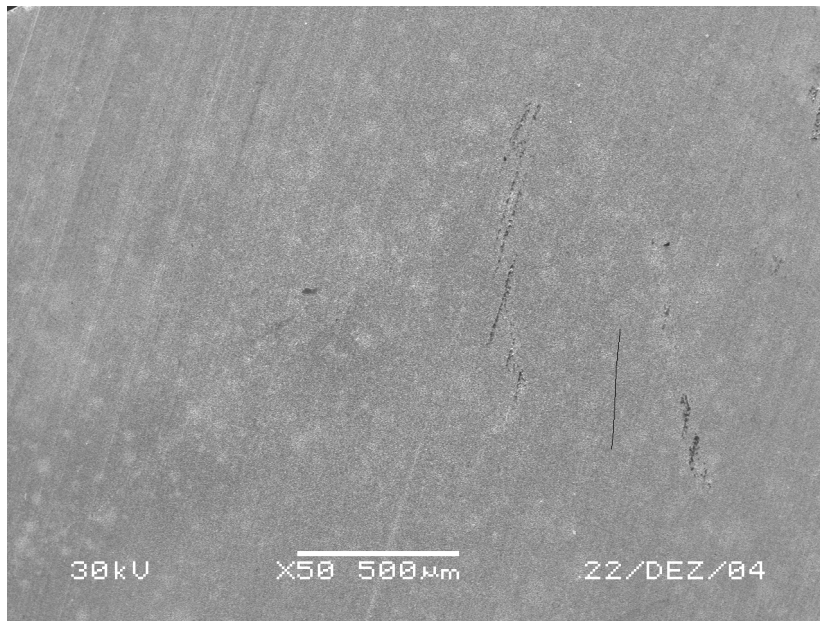


Figure 6 : Surface of hard metal after immersion in a 10 g  $K_3 [Fe (CN)_6]$  + 10 g KOH + 100 ml  $H_2O$  and then in a 6  $H_2O$  + 3 HCl + 1  $H_2O_2$  mixture.

Figure 7 shows the surface of a hard metal sample after immersion in a 10 g  $K_3 [Fe (CN)_6]$  + 10 g KOH + 100 ml  $H_2O$  and then in a 3 ml  $H_2SO_4$  + 88 ml  $H_2O_2$  mixture. In this case, the surface is less affected, less rough, and less porous. The HCl based acid reacted more with the sample than the  $H_2SO_4$  based solution did.

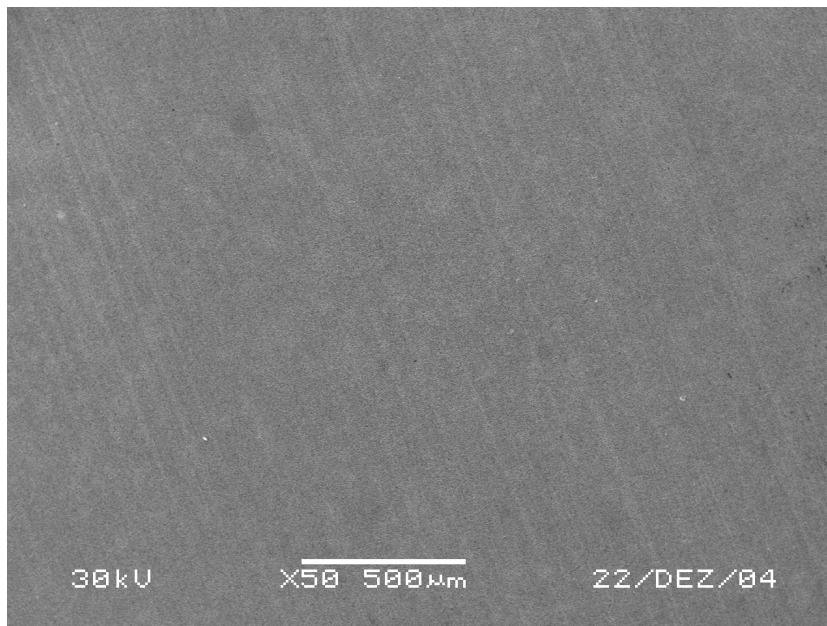


Figure 7 : Surface of hard metal after immersion in a 10 g  $K_3 [Fe (CN)_6]$  + 10 g KOH + 100 ml  $H_2O$  and then in a 3 ml  $H_2SO_4$  + 88 ml  $H_2O_2$  mixture.

Then diamond powder was dispersed over the samples and the films were deposited as described in the “Experimental” section. The samples which had been prepared with the 10 g  $K_3 [Fe (CN)_6]$  + 10 g KOH + 100 ml  $H_2O$  mixture had no adhesion problems, while for the samples which had been prepared with the pure KOH base, or which had only passed through the acid solutions, the films peeled off, once the samples left the reactor. First adhesion tests showed that the HCl based acid solution ( after preparation in the 10 g  $K_3 [Fe (CN)_6]$  + 10 g KOH + 100 ml  $H_2O$  mixture) resulted in the diamond film with the best adhesion characteristics.

The probable reason that the KOH treated samples did not perform as well as the other samples is probably that the KOH did not open the tungsten carbide as much as the other mixture and hence the cobalt was not etched away from as far under the surface. Because of the high temperature and long duration of the diamond deposition, the cobalt diffuses from inside the sample towards the surface. When the tungsten carbide was not enough depleted from cobalt, diamond adhesion was bad. There are strong indications that the combination of the 10 g  $K_3 [Fe (CN)_6]$  + 10 g KOH + 100 ml  $H_2O$  mixture with the HCl based acid removed the cobalt also from far under the surface, leaving a relatively thick tungsten carbide layer depleted from cobalt. During deposition, cobalt was not able to reach the surface and therefore the final adhesion of the deposited diamond film was very good.

The final structure of this film is shown in figure 8. The continuous, polycrystalline film adheres well to the substrate. This kind of films may be used to extend the mechanical characteristics of hard metals for different mechanical operations.

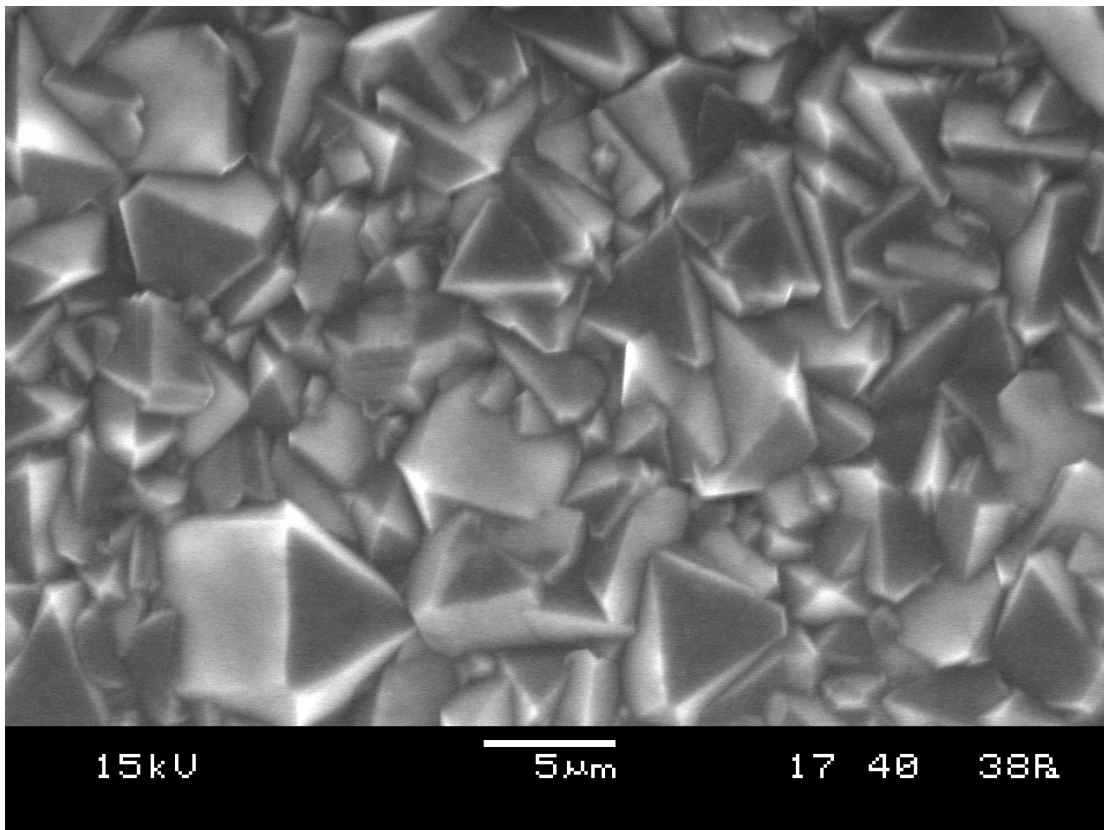


Figure 8: Diamond film deposited on hard metal sample after preparation of the surface by immersion in a 10 g  $K_3 [Fe (CN)_6]$  + 10 g KOH + 100 ml  $H_2O$  and then in a 6  $H_2O$  + 3 HCl + 1  $H_2O_2$  mixture

#### 4. CONCLUSIONS

Different procedures to prepare hard metal samples (WC – 6% Co) for polycrystalline diamond deposition were investigated. These preparations have to remove cobalt from the surface and even from nearby the surface. Pure KOH and the 10 g K<sub>3</sub> [Fe (CN)<sub>6</sub>] + 10 g KOH + 100 ml H<sub>2</sub>O mixture were used as a first step to break the tungsten carbide structure open. EDX spectra showed that both solutions accomplished this task, but that the 10 g K<sub>3</sub> [Fe (CN)<sub>6</sub>] + 10 g KOH + 100 ml H<sub>2</sub>O was most successful. Afterwards several samples were etched in three different acid solutions, based on chlorine, nitric and sulphuric acid. The EDX spectra showed that all these solutions managed to remove well the surface cobalt. SEM analysis showed that the 10 g K<sub>3</sub> [Fe (CN)<sub>6</sub>] + 10 g KOH + 100 ml H<sub>2</sub>O solution modified the hard metal surface dramatically, turning it spongy and porous. Then a diamond film was deposited. All the samples which had passed through the 10 g K<sub>3</sub> [Fe (CN)<sub>6</sub>] + 10 g KOH + 100 ml H<sub>2</sub>O solution showed good adhesion, all the others suffered peeling. The HCl based acid solution promoted the best adhesion of the diamond film on the hard metal samples. The resulting films and samples are very promising for mechanical processes such as drilling, milling etc.

#### 5. ACKNOWLEDGEMENTS

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## **ANALISE DE DIFERENTES PROCEDIMENTOS DE PREPARAÇÃO DE SUPERFÍCIES PARA A DEPOSIÇÃO DE REVESTIMENTOS DE DIAMANTE POLICRISTALINO SOBRE METAIS DUROS**

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**Resumo:** *carbonetos cementados ou "metais duros" apresentam diversas aplicações interessantes como ferramentas e mesmo como elementos de máquinas na indústria mecânica, por exemplo: ferramentas de conformação ou usinagem de metais., bem como mineração. Dentre outras composições: a combinação carboneto de tungstênio (WC) com 6% de cobalto como fase ligante é considerada como a composição com o balanço mais adequado entre tenacidade e propriedades tribológicas (resistência a abrasão e a aderência). Uma melhora nas propriedades tribológicas pode ser alcançada pelo revestimento destas ferramentas com um filme fino de diamante sobre o topo de sua microestrutura que contacta o material que está sendo processado, ampliando bastante a vida destas ferramentas. Uma preocupação especial aqui é o teor de cobalto, ou ainda a composição química da fase ligante no substrato que induz a uma aderência ruim do filme de diamante. Tratamentos especiais foram necessários para remover o cobalto da superfície, bem como uma combinação de imersão em uma solução de 10 g K<sub>3</sub>[Fe(CN)<sub>6</sub>] + 10 g KOH + 100 ml H<sub>2</sub>O e então imersão em uma solução de 6 H<sub>2</sub>O + 3 HCl + 1 H<sub>2</sub>O<sub>2</sub> m que resulta em um excelente aderência do filme depositado a seguir.*

**Palavra-chave:** *revestimento duro, diamante, metal duro, revestimento superficial.*