

# ANALYSIS OF TiC-BASED ALLOYS PREPARED BY MECHANICAL ALLOYING AND SPARK PLASMA SINTERING

Mohsen Mhadhbi | National Institute of Research and Physical-Chemical Analysis | Ariana, Tunisia  
 Frédéric Schoenstein | Paris 13 University | Sorbonne Paris City, France  
 Noureddine Jouini | Paris 13 University | Sorbonne Paris City, France

## Introduction

The majority of the products composed of TiC-based alloys are fabricated according to traditional powder metallurgical technology like preparation of a mixture of TiC powder with metal binder, hot isostatic pressing, compaction of the mixture, forging, sintering and additional treatment [1]. Thus, to synthesis a homogeneous titanium carbide (TiC) and binder metal mixture, mechanical milling of the mixture is used. During this process, the physical, chemical, and mechanical properties of the starting components undergo changes. This influences the structure and properties of hard TiC-based alloys. However, the character of the interaction between the binder metal and titanium carbide effect the mechanical properties of TiC-based alloys [2]. These materials are used in several applications like biomedical, aircraft, cutting tools, etc.

Our work aims to analyse the proprieties of TiC-based alloys prepared by mechanical alloying followed by spark plasma sintering.

## Experimental

Elemental powder mixtures of Ti (< 40µm, 99.9%), W (1-5µm, 99.9%), Zr (99.9%), Cr (99.9%), and carbon (99.9%) were sealed into a stainless steel vial with 5 stainless steel balls (15 mm in diameter and 14g in mass) in a glove box filled with purified argon. The ball to powder weight ratio was 70:1. The mechanical alloying (MA) process was performed at room temperature using a high energy planetary ball mill (Fritsch, Pulverisette 7). Thus, the powders were milled for 20h. The obtained nanocrystalline powders were sintered using SPS-5155 SYNTEX apparatus under a pressure value of 80MPa for 5 min. Before analysis, the obtained samples were polished using a polisher model PX 300. The microstructure and the morphology of the samples were studied by using X-ray diffraction (XRD) using X'Pert PRO MPD PANALYTICAL apparatus and scanning electron microscopy (SEM) using an FEI Quanta 200 environmental scanning microscope. The porosity of the samples was determined by using IA44 apparatus. Therefore, the Vickers hardness of the samples was measured by LV hardness tester. The results show that the samples have homogenous structure with a low porosity, a high relative density, and a high Vickers hardness.



Planetary ball Mill      SPS-5155 SYNTEX apparatus      Final product (bulk)

## Results and Discussion

The synthesized powders obtained by milling and sintering are analyzed by various techniques.

### X-Ray Diffraction analysis

Figure 1 illustrates the XRD patterns of the powders milled for 20h and sintered for 5 min.

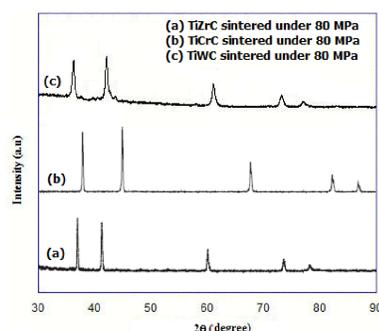


Figure 1. XRD patterns of TiC-based powders: (a) TiZrC sintered under 80MPa, (b) TiCrC sintered under 80MPa, and (c) TiWC sintered under 80MPa

- Nanocrystalline phases were obtained for all the powders milled for 20h.
- The mean crystallite size, determined by the FullProf program using the Rietveld refinement, decreases with increasing milling time, on the other hand the mean microstrain increases with increasing milling time.

### Scanning Electron Microscopy analysis

The SEM micrographs of the samples (powders) milled for 20h and sintered for 5 min are represented in the following figure 2.

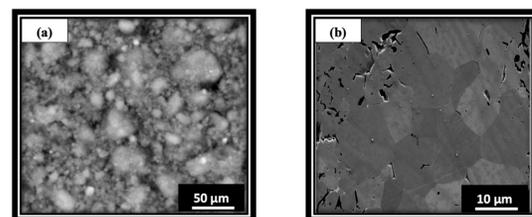


Figure 2. SEM micrographs of TiC-based powders: (left) milled for 20h and (right) sintered for 5 min

- By increasing the milling time up to 20h, the morphology of powders is still homogenous but with very fine agglomerated particles smaller than 1 µm in size.
- After sintering for 5 min, small pores are observed. In fact, the intergranular pores coalesced to form closed pores. Particles of different sizes agglomerate to form coarse grains, while heterogeneous distribution of equiaxed grains still occurs.
- Sintering process leads to the grain growth of the powder particles with retention of nanoscale.

## Physical and Mechanical Properties

The experimental results including mechanical and physical properties are illustrated in Table 1.

TABLE 1: RELATIVE DENSITY, POROSITY, AND VICKERS HARDNESS OF PREPARED MATERIALS			
Material	Relative density (%)	Porosity (%)	Vickers Hardness (HV)
TiWC	98	5	2980
TiZrC	98	4	2765
TiCrC	97	5	2350

- The materials have an excellent physical and mechanical properties.
- Dense materials are obtained.
- The obtained materials are expected to replace the standard materials.

## Conclusion

In this work, bulk nanocrystalline carbides were fabricated through mechanical alloying followed by spark plasma sintering. The mean crystallite size reach about 10 nm after 20h of milling. The increasing of milling time decreases the temperature of densification. The sintered materials show excellent physical and mechanical properties. The density of the final products is 98% and the hardness is about 2800HV, which is greater than that of TiC carbides (2200HV). The maximum hardness was obtained for the more dense materials, meanwhile, the grain size is large. The hardness evolution doesn't follow the Hall-Petch behavior and is more affected by the material porosity.

[1] F. Eisenkolb, Powder Metallurgy, Technik, Berlin, 1955.

[2] D. Moscovitz and H.K Plummer, Proc. Int. Conf., Sci. Hard. Mater., No. 4, London, 1983, Jackson, pp. 299-309.