11th International Conference on Advanced Materials
Rio de Janeiro Brazil
September 20 - 25
ICAM2009

Production and Characterization of nanoparticles of the system Ti-N-O produced from Vapor Phase

A. V. Grillo(1)*, B. C. Di Lello(2), F. J. Moura(1) and I. G. Solórzano(1)

(1) DCMM, Pontifícia Universidade Católica do Rio de Janeiro, e-mail: aleflugrillo@gmail.com
(2) UNESA, Universidade Estácio de Sá, Rio de Janeiro, Brazil
* Corresponding author.

Abstract – The importance of titanium nitride is particularly due to its low friction coefficient, extreme hardness, high thermal stability, metallic conductivity and extremely high resistance to chemical attack and non-toxicity. Therefore used as a coating material in the mechanical industry, related to thermal management in microelectronics, medical and structural applications. The objectives of this work are the production of TiN powders from vapor phase reaction between titanium tetrachloride ($TiCl_4$) and ammonia (NH_3) in a tubular reactor, and the study of the effects of the experimental parameters on mean particle size. Besides powder production, TiN was deposited in layers on the internal walls and internal structures of the reactor. The microscope analysis of the layers showed a columnar hexagonal crystal growth with highly regular morphology.

Nitrides, especially those of transition metals, have received increasing attention in recent years because of their unique chemical and physical properties. The importance of nitride titanium (TiN) is due to its properties of high thermal conductivity, chemical inertial, hardness, wear resistance, high electrical conductivity and nontoxicity [1]. The objective of this work is the production of TiN powder from vapor phase reaction between titanium tetrachloride ($TiCl_4$) and ammonia (NH_3) in a tubular reactor and the study of the effects of the reaction parameters, temperature and space time, on powder particle size. The experimental results showed that the variation of these parameters produced nanoparticles of TiN with different heterogeneous sizes. Besides in the production of titanium nitride powder, there was also the presence of ammonium chloride (NH_4Cl), co-product of the nitridation reaction. During the handling of the TiN powder occurred the formation of TiO_2 due to its reaction with air.

The dynamic system consisted of on a quartz tube reactor with controlled injection of ammonia and argon. $TiCl_4$ vaporization set up and powder collection systems are presented in the Figure 1. Titanium tetrachloride ($TiCl_4$) in the liquid phase is vaporized at a temperature of approximately 80°C. The injection of argon gas (Ar) carried out the $TiCl_4$ vapor formed to the central part of the reactor. During the process of synthesis, it was observed that in the colder areas of the reactor, deposits of ammonium chloride (NH_4Cl), by co-product was formed, besides, it was also observed the presence of titanium dioxide (TiO_2), anatase, formed from the oxidation of TiN with the air.

The samples analyzed was obtained at 900°C using argon flux of 0,54L/min and 0,57 L/min of ammonia. X ray diffraction and Transmission Electron Microscopy has been used as characterization tool. The former confirmed the presence of TiN, TiO_2 and NH_4Cl (Fig. 2). The later allowed the observation of a good dispersion of particles with spherical and faceted morphologies. The powder particles were observed to be in the 10-15nm range (Figure 3). In this study the experimental results showed that the variation of parameters of TiN with different sizes, heterogeneous, with sizes between 5nm-35nm. Current studies focus on the control of particles size and its N/O ratio.

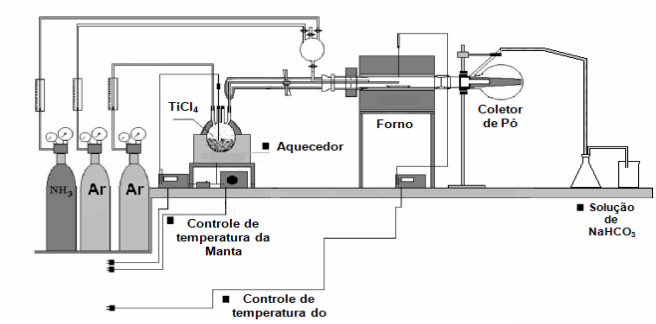

Figure 1: Schematic diagram of the equipment used to produce TiN from vapor phase.

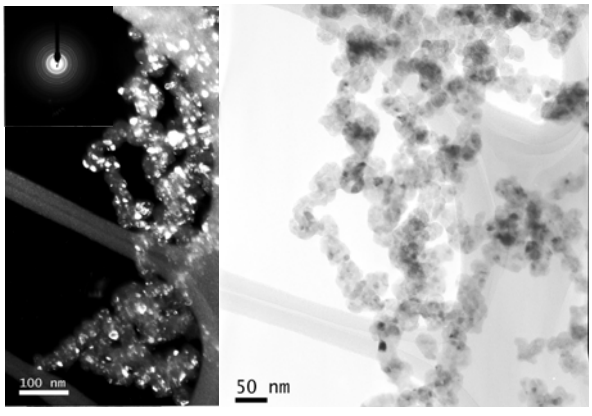

Figure 3: TEM of powder nanoparticles of TiN **a)** Dark field and diffraction pattern images **b)** Bright field image.

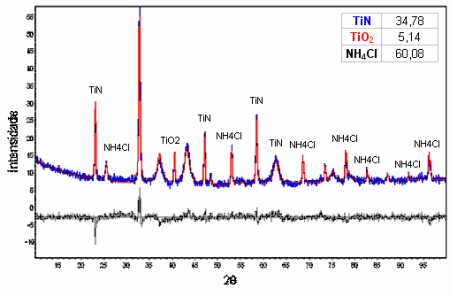

Figure 2: XRD of the powder. The formation of TiN was confirmed.

References

[1] Q. Guo, Y. Xie, X. Wang, S. Lv, T. Hou and C. Bai. J. Am. Ceram. Soc. 88 [1] 249-251.