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Synthesis and characterization of aluminum titanium carbonitride TiAlCN via mechanical alloying

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This study presents synthesis of titanium aluminum carbonitrides alloy (TiAlCN) by mechanical alloying in Attritor ball mill from elemental powders of titanium, aluminum and graphite in nitrogen atmosphere. TiAlCN was characterized by SEM, XRD, DSC and FTIR techniques. XRD showed presence of titanium aluminum nitride, aluminum nitride, titanium carbide and titanium aluminum carbon nitride, while SEM showed existence of micro and nano particles with high agglomeration. Energy-dispersive spectroscopy (EDS) analysis shows a homogeneous distribution of elements, and mapping analysis from X-rays confirms distribution of elements.

Keywords: Carbides, Elemental powders, Mechanical alloying, Nitrides, Titanium aluminum carbonitrides alloy

Introduction

Development of Ti-Al-N ternary alloy gives an increase in hardness and oxidation resistance in comparison with binary alloy TiN[1]. Quaternary alloys type, titanium aluminum carbonitride titanium (Ti-Al-N-C) has been produced by pulsed laser deposition technique[2-3], magnetron sputtering technique[6], and chemical depositions techniques[4]. Titanium aluminum carbonitrides alloy (TiAlCN) hard coating shows high wear and erosion resistance, under high cutting velocity and without lubrication[6]. This study presents synthesis by mechanical alloying and characterization of powder alloy TiAlCN as target to obtain hard coatings.

Experimental Section

TiAlCN powder (Ti, 55.1; Al, 31.0; and C, 13.9%) was obtained by mechanical alloying in a ball milling atritor of vertical impeller[6]. A stainless steel vial and various Cr steel balls (diam, 6-8 mm) were used. Elemental powder contained: pure Ti (particle size, 150 μm), 99.7 wt%; Al (particle size, < 200 μm), 99.95 wt%; and C (particle size, <45 μm), 99.99 wt%. Mill container air was evacuated using a vacuum pump and then filled with nitrogen gas. Mass balls and powder ratio was 70:1, with 500 rpm constant rotational velocity for 140 h. Compaction process was carried out for specimen 1 with 70 h milling, and specimen 2 with 140 h milling using a universal compression machine, which has a 2000 KN loud cell and a compacting matrix that allows obtaining targets (diam, 1.2 cm). Powder material was compacted (as specimens 1 and 2 of 1 g each), by applied pressure (800 MPa), and target did not present visible defects and was easily ejected from die.

Sintering was realized in Nabertherm furnace model LHT 02/1. Specimen 1 was sintered at 1000°C for 1 h with a heating rate of 5°C/min, and specimen 2 at 1400°C for 2 h with a heating rate of 10°C/min. Sintering at 1400°C target showed cracks in bulk in many pieces. Sintering at 1000°C target showed a light delamination, indicating phase transformation between 25-1000°C, after having used differential calorimetry scanning (DSC) to corroborate.

X-Ray diffraction analyses (XRD) was carried out in a Panalytical reference X'Pert PRO MPD with Cu ceramic and solid state detector reference PixCel. Microstructure of milled powder, Energy-dispersive spectroscopy (EDS) and mapping of elements was studied in a SEM JEOL JSM 6490LV Scanning Electron Microscope (SEM). Samples transition temperatures were measured by a TA Instruments SDT Q600 Differential Scanning Calorimetry (DSC) Analyzer. Powders were heated to1000°C at a rate of 10°C/min, and Fourier Transform Infrared Spectroscopy

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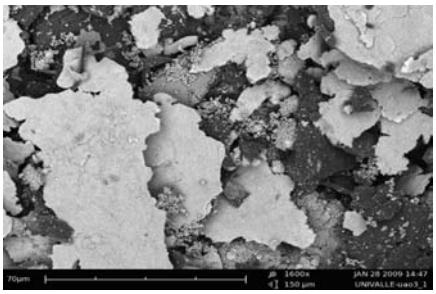

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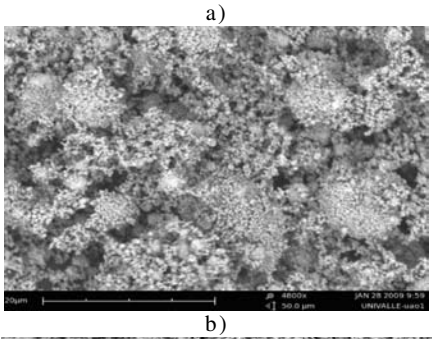

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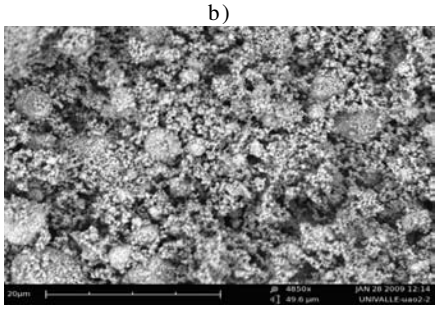

c)

Fig. 1—SEM images; a) Powder without milling process, 4800X; b) Specimen 1 with 70 h of milling, 4850X; and c) Specimen 2 with 140 h of milling, 4850X

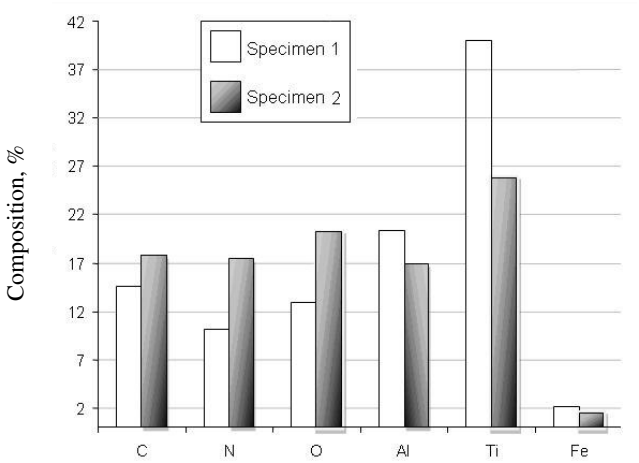

Fig. 2—Bar diagram of average quantitative analysis of specimens 1 and 2

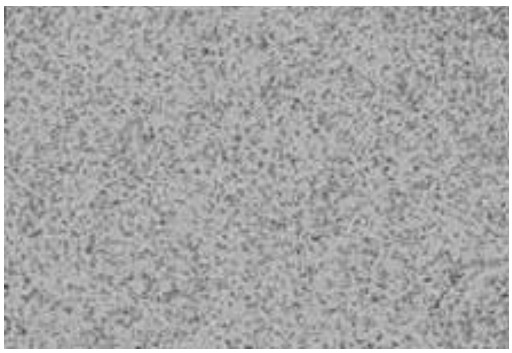

Fig. 3—X-ray map SEM of Ti in specimen 1

(FTIR) analysis was done in Thermo Electron Corporation IR 200 Spectrometer.

Results and Discussion

Al, Ti and C powders were manually mixed for better homogenization of elements. Laminar, flakes, and porous particles of Ti, Al and C were displayed with maximum size of 35 μm (Fig. 1). Specimen 2 with 140 h of milling (Fig. 1c) showed more homogeneity than specimen 1 with 70 h of milling. Clusters and porous particles, showed a particle size of 1-5 μm. EDS micro-analysis was located on 3 different places in each specimen, with 70 h and 140 h milling time respectively (Fig. 2). Under X-ray maps realized with SEM, for specimens 1 and 2 (Fig. 3), Ti Al, C and nitrogen elements were found homogeneously distributed in surface tested; Ti showed more homogeneity. XRD patterns milled powders showed (Fig. 4) presence of titanium aluminum nitride, aluminum nitride, titanium carbide, aluminum titanium and carbon nitride. Refined XRD results indicated that in case of specimen 1 with 70 h of milling time, carbon nitride has highest content of total elements (54.5%); other elements present were aluminum titanium (15.2%), titanium aluminum nitride (12.1%), aluminum nitride (10.1%) and titanium carbide (8.1%). In case of specimen 2, with 140 h of milling, refining of XRD patterns showed presence of carbon nitride (57.6%), aluminum titanium (16.2%), titanium aluminum nitride (14.1%), titanium carbide (9.1%) and aluminum titanium nitride (3.0%).

DSC curves present phase transformation at 417°C and 423°C for 70 h (Fig. 5a) and 140 h (Fig. 5b) respectively, confirming that cracking in sintering process is due to heating rate, 10°C/min, from 25°C to 1400°C, in comparison to heating rate 5°C/min, from 25°C to 1000°C whose specimen do not show fracture, only light delamination. FTIR spectrum (Fig. 6) demonstrates presence of compounds containing Al, C, N, O-H in both specimens, corresponding to 70 h and 140 h of milling time. Bands at 3800 and 2900 cm^{-1} corresponds to O, N and H atoms[7-8], while peak between

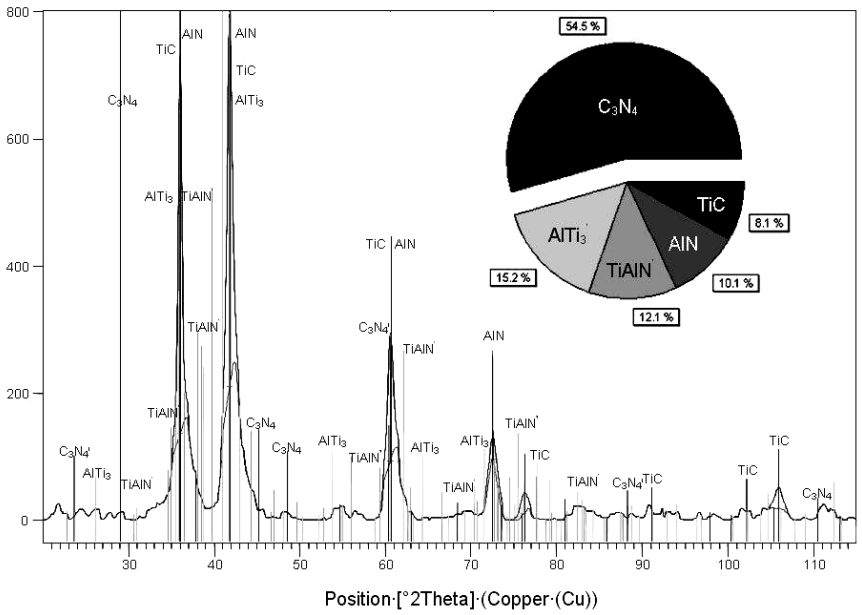

Fig. 4— XRD spectrum of elements present in alloy, 70 h of milling

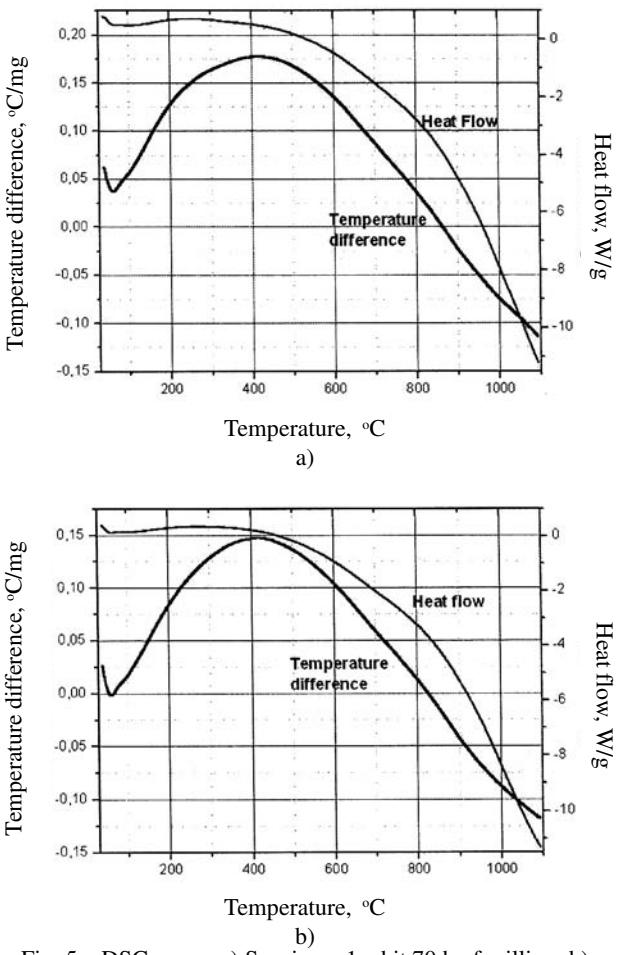

Fig. 5—DSC curve; a) Specimen 1 whit 70 h of milling; b) Specimen 2 with 140 h of milling

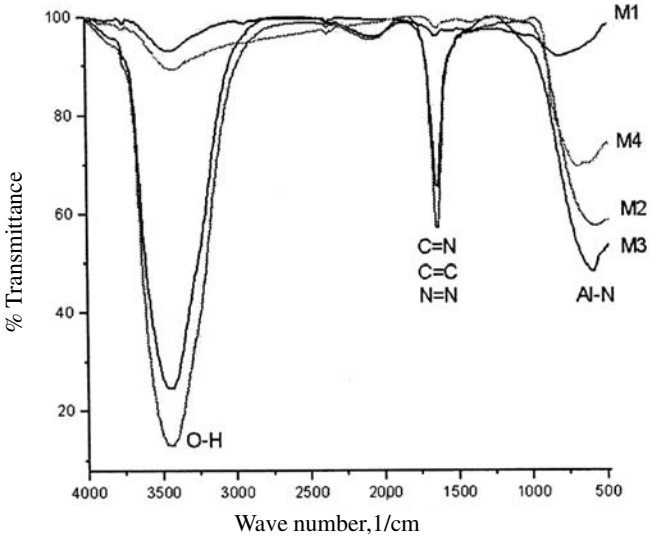

Fig. 6—FTIR spectrum of powder specimen analyzed at different temperatures of sintering (M1, alloyed material with 70 h of milling; M2, alloy material with 140 h of milling; M3, alloyed powder with 70 h and sintering at 1000°C; and M4, alloyed powder with 70 h and sintering at 1400°C)

1630 and 1645 cm^{-1} corresponds to double bonds of C=N, C=C and N=N. A band located at 600, 950 and 980 cm^{-1} is associated with Al-N phases[9]; a wide band located between 1300 and 1700 cm^{-1} is correlated with graphite[10].

Conclusions

Powders processed by mechanical alloying with 70 h and 140 h of milling, were analyzed with FTIR and

XRD characterization techniques. Presence of carbon nitride, titanium aluminum, titanium aluminum nitride, aluminum nitride and titanium carbide compounds was found. SEM images showed evolution of particle size in initial state, 70 h and 140 h of milling time, confirming size reduction from 35 μm to 1 μm in final state of milling. EDS and mapping analyses present a successful elements homogenous distribution of TiAlCN alloys. EDS showed a phase transition close to 420°C; therefore it is suggested that sintering process is to be made with a heating rate over 10°C/min in order to avoid phase transition.

Acknowledgments

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