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TiTaCN-Co cermets prepared by mechanochemical technique: microstructure and mechanical properties

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Abstract

Microstructure and mechanical characterization of (Ti,Ta)(C,N)-Co based solid solution cermets prepared by two mechanochemical synthesis processes (one- and two-step milling) and a pressureless sintering in protective helium atmosphere. Materials with composition of $Ti_xTa_{1-x}C_{0.5}N_{0.5}$-20%Co with two different Ti/Ta ratios (x = 0.9 and x = 0.95) were developed to prepare four groups of experimental materials. Microstructures were observed using confocal microscopy and grain size was evaluated using image analysis. Mechanical testing was carried out using nanoindentation equipment and nanohardness and indentation Young´s modulus were obtained. Mechanical properties of individual phases were measured using single load/unload method with 20 mN maximum load (40 mN/min loading rate and maximum 10 s holding time for each indent). Mechanical properties of each material as a bulk were obtained also by single load/unload method with 300 mN maximum load (600 mN/min loading rate and maximum 10 s holding time). The resulting mechanical properties were comparable to that of typical industrial hardmetal cermets. Two-step milling resulted in finer microstructure but a wider range of grain size distribution. No significant dependence between mechanical properties and number of milling steps was found. However, the materials with higher amount of Ta showed slightly higher indentation elasticity modulus.

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Keywords: cermet; hardmetal; mechanochemistry; hardness, indentation

1. Introduction

Modern hardmetals used to machine demanding materials such as steels with high strength and/or hardness, complex alloys, and similar, rely on very hard, usually some sort of ceramic, particles placed inside or bond together by metal based matrix, often some solid solution. A typical example is WC-Co cermet, used for cutting, machining, and other type of tools. Hard components there are made of tungsten carbide with very high hardness and excellent abrasion resistance [1]. The soft Co based binder provides sufficient fracture toughness. Suitable stiffness can in the WC-Co be controlled by the binder content - less Co makes the composite stiffer and vice versa [2]. Recently there has been an effort to replace the tungsten based materials with other alternatives. Metal-ceramic composite materials made from Ti(C, N) are currently being introduced as cutting tool materials because of their good lifetime, slow wear and good chemical stability at high temperatures [3,4]. Titanium carbonitride, Ti(C,N), is a solid solution of the TiC-TiN system. It has high melting point, high thermal and electrical conductivities, good thermal and chemical stability [5-7]. These materials are potentially lighter and more environmentally friendly than WC-Co. A major problem in the use of metal-ceramic composite materials of Ti(C, N) type as replacement for the WC-Co in large scale industrial market is their lower strength. This lower strength is attributed to the ceramic particles. It can be improved at the expense of either hardness, which increases with the increasing volume fraction of hard particles, a reduction in size of these grains and the distance between them. Heavy metals improve the surface quality allowing excellent cleavage resistance, machinability and

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dimensional accuracy of the workpiece. Regarding the hard grains, their core is usually composed of dissolved Ti (C, N) particles in the original pre-sintered metal-ceramic composites [8]. Boundary phase is formed during the liquid phase sintering when the complex carbonitride solid solution containing titanium and other transition metals precipitates from the supersaturated binder phase, which consists of Ni, Co or their alloys and undissolved core particles. The transition between the core and the edge can generate residual stresses that promote cracking and consequent reduction in toughness of materials [8]. For further improvement of their properties, other binary carbides (e.g. NbC, TaC, Mo_2C) are usually added. TaC is often used to enhance stability at higher temperatures, hardness, thermal shock and creep resistance [3,9,10].

Mechanochemistry is an interesting method of chemical synthesis in solid state materials. It usually uses high energy milling [11-13]. It is potentially cheap and easily scalable technique where dissolution or melting of the reactants is not required.

The aim of this work was to prepare a series of alternative hardmetal systems by means of high energy milling and to characterize them from the point of view of microstructure, hardness and modulus of elasticity.

2. Experimental procedure

2.1. Materials

The experimental materials were prepared by high energy milling which utilizes mechanically induced self-sustaining reaction (MSR) from commercially available pure powders [9,14,15]. The starting materials were: Titan powder (purity 99%, grain size < 44 µm, StremChemicals); Tantalum powder (purity 99,6%, grain size < 44 µm, Alfa-Aesar); Graphite powder (grain size < 53 µm, Fe ≤ 0.4%, Merck); Cobalt powder (purity 99.8%, grain size < 150 µm, StremChemicals).

Milling and homogenization was realized in a modified planetary ball mill (Planetary Mill Pulverisette 4, Fritsch). 46.5 g of an elemental powder mixture together with tempered steel balls (15, d=20 mm, m=32.6 g) used as milling medium were placed in a 300 ml tempered steel vial (67Rc) and milled under 0.6 MPa of high-purity nitrogen gas (H_2O and $O_2 \leq 3$ ppm, Air Liquide) The powder-to-ball mass ratio (PBR) was ~1/10.5, and a spinning rate of 400 rpm for both the rotation of the supporting disc and the superimposed rotation in the direction opposite to the vial was employed. High energy input activated chemical processes and that lead to MSR and consequently to creation of titanium and tantalum carbonitrides.

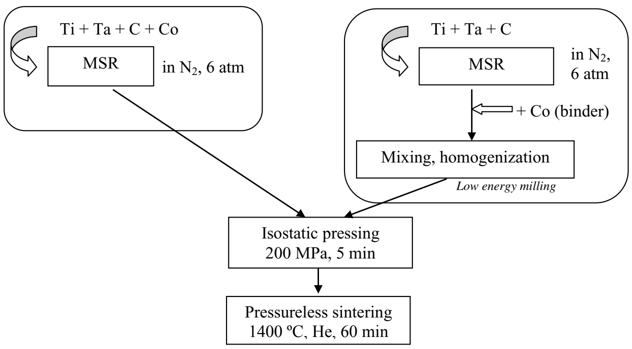

Fig. 1. Schematic of preparation of experimental materials – one step (left) and two-step milling (right).

The final mixtures for sintering were prepared by two ways – one step and two step millig, Fig. 1. In one step milling all starting powders were milled together from start together with cobalt binder. In two-step milling first Ti, Ta, and C powders were milled in nitrogen atmosphere, and only after the MSR had taken place and (Ti,Ta)(C,N) had synthesized, the Co powder was added.

The prepared mixtures were isostatically pressed at 200 MPa for 5 mins. Then they were pressurelessly sintered at 1400 ºC for 60 min in protective He atmosphere.

Resulting samples were discs of 20 mm diameter and height around 5 mm. Four different experimental states of $Ti_xTa_{1-x}CN$-20%Co were prepared with two different Ti/Ta ratios and two ways of milling, as it is summarized in Tab. 1.

Table 1. Experimental materials: notation, composition, milling type.

Material	Ti / Ta ratio	Milling
90/10-1	0.9 (Ti) / 0.1 (Ta)	1 step
90/10-2	0.9 (Ti) / 0.1 (Ta)	2 steps
95/5-1	0.95 (Ti) / 0.05 (Ta)	1 step
95/5-2	0.95 (Ti) / 0.05 (Ta)	2 steps