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Electric current assisted sintering of continuous functionally graded Ti_2AlN/TiN material

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Abstract

A Ti/AlN powder mixture with a molar ratio of 2:1 was consolidated by electric current assisted sintering (ECAS) using a special designed graphite die which can give rise to a huge temperature gradient along the vertical direction in the sintering compact. By this way, a continuous microstructural evolution during the formation of Ti_2AlN was observed. At 1200 °C, a Ti_2AlN–TiN composite was prepared through the in situ decomposition of Ti_2AlN. The SEM and XRD results revealed that a continuous variation of Ti_2AlN and TiN phase content was achieved. The gradual hardness distribution across the entire composite further indicated that a continuous functionally graded Ti_2AlN/TiN material was obtained rapidly by taking advantage of the ECAS method.

Keywords: B. Composites; C. Hardness; D. Nitride; E. Structural application

1. Introduction

In order to reduce the stress concentration in a component when it suffers rigorous conditions such as rapid heating or cooling and sudden impact, a transitional layer between different materials is usually introduced. This gives the birth of the functionally graded materials (FGMs) [1]. The FGMs are usually composites in which the composition and/or microstructure vary gradually along one, two or three dimensions [2]. There are numerous methods to process FGMs, such as casting [3], laser cladding [4], combustion synthesis [5] and powder metallurgy [6]. Among these, the powder metallurgy technique is one of the most important ways since it can be easily accomplished by stacking material ingredients into layers through a variety of methods [1,2]. However, the partial sintering of different layers may compromise the reliability of the component under operating conditions since layers with different compositions need different temperatures to be fully densified. Therefore, to overcome this problem, new approaches must be considered.

Over the last decades, electric current assisted sintering (ECAS), also known as spark plasma sintering (SPS), has been extensively used for synthesis or consolidation of various materials. The rapid heating rate and possible plasma during ECAS make it possible to fabricate materials at low temperature and with short periods of time. Although a temperature gradient always exists inside the die and specimen due to the intrinsic heating method [7,8], it can be minimized by the careful process, such as using the highly symmetrical heating set-up together with adiabatic materials around the die and taking the multi-stepped heating rate [9,10]. In the most cases, the temperature gradient is minimized in order to prepare homogeneous materials. However, from a different point of view, the temperature gradient can be enlarged if the heating set-up departs from the high symmetry. Based on this idea, several attempts have been done to fabricate the FGMs by designing the heating set-up [11–15]. For example, Hong et al. [12] prepared ZrB_2–SiC/ZrO_2(3Y) FGM by using an unsymmetrical graphite die. In that work, every layer with the different contents of the ZrB_2 and SiC/ZrO_2(3Y) was fully densified because of the temperature gradient. Compared with Hong's work, Hulbert et al. [13,14] fabricated the B_4C/Al FGM with a continuous gradation of microstructure and thus performed increased mechanical properties. Lately, Belmonte et al. [15] used temperature gradient to control the phase transformation of Si_3N_4. They achieved a continuous gradient of α/β phase contents and grain sizes from a sole homogeneous

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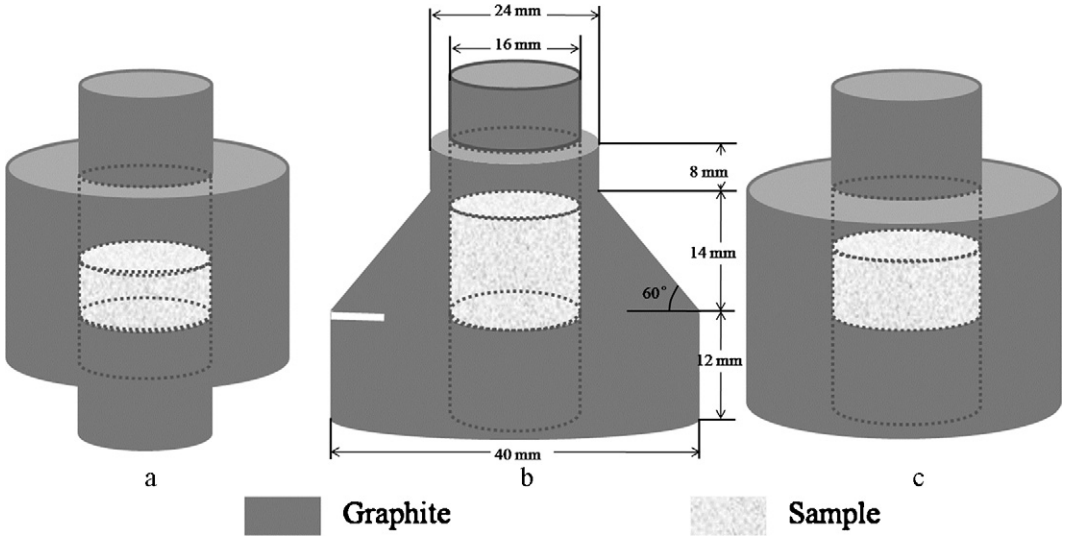

Fig. 1. Graphite moulds with three different shapes used in ECAS. (a) For homogeneous temperature distribution; (b) and (c) for gradient temperature distribution.

starting powder. The possibility of designing a temperature gradient into the material during sintering opens a new window for synthesizing FGMs by ECAS.

The Ti_2AlN/TiN FGM is considered as a promising composite to be used in high-strength armour and high-abrasive conditions due to the high strength and stiffness of the TiN phase combined with the tough and ductile Ti_2AlN [16,17]. However, to our best knowledge, few studies on the preparation of Ti_2AlN/TiN FGM by any method have been reported. In the present work, the Ti/AlN powder mixture was adopted to synthesize the Ti_2AlN/TiN FGM by ECAS using a special designed graphite mould and the continuous microstructural evolution during the reaction sintering of Ti and AlN was studied.

2. Experimental

Commercial Ti (particle size: 25 μm; purity: 99.5%) and AlN (particle size: 3 μm; purity: 99.2%) powders were used as raw materials. The powder mixtures, having a stoichiometric Ti to AlN molar ratio of 2:1, were ball-milled for 12 h in an air-sealed container with a ball to powder mass ratio of 4:1. The powder mixture was then dried, sieved (100 mesh) and filled into the designed graphite mould which is shown in Fig. 1(b). The subsequent sintering was carried out in the vacuum of 6 Pa by the ECAS apparatus (ED-PASIII, Elenix). A heating rate of 100 °C/min and a uniaxial pressure of 30 MPa were applied. The temperature was regulated and controlled by an infrared pyrometer that was focused on the hole labelled on the graphite die. In order to investigate the reaction between Ti and AlN, the powder compacts were heated to 900 °C, 1000 °C, 1100 °C and cooled down immediately with the cooling rate >200 °C. Finally, the Ti_2AlN/TiN FGM was prepared at 1200 °C with holding time of 3 min. The sintered samples had diameters of 16 mm and heights of 10 mm. In order to eliminate the graphite contamination, the surfaces of the samples were ground down 1 mm.

Before characterization, the samples were cut into small pieces with different shapes by electric discharge machining (EDM). The surfaces of the pieces were polished down to 1 μm, finishing by diamond paste. The X-ray diffraction (XRD) was conducted on the polished surface with a PANnalytical X'Pert, using Cu $K_{\alpha 1}$ radiation at 45 kV and 40 mA. The microstructures were observed by Scanning Electron Microscopy (SEM, S-2700) and Optical Microscopy (OM, MEF-3). For the OM, the polished pieces were etched with an acidic solution (HNO_3:HF:H_2O = 1:1:2). The hardness across the sample was tested by Vickers indentation method using an external force of 9.8 N and holding for 15 s. Five indents were taken at the equivalent position.

Fig. 2. Real-time colour distribution of graphite mould during the sintering. Note that the white, yellow and red colour represent high, intermediate and low temperature respectively. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of the article.)