



Contents lists available at www.sciencedirect.com

Journal of the European Ceramic Society

journal homepage: www.elsevier.com/locate/jeurceramsoc



High temperature properties of the monolithic CVD β -SiC materials joined with a pre-sintered MAX phase Ti_3SiC_2 interlayer via solid-state diffusion bonding

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ARTICLE INFO

Article history:

Received 17 August 2016
Received in revised form 2 November 2016
Accepted 4 November 2016
Available online xxx

Keywords:

Diffusion joining
SiC
MAX phase
High temperature mechanical properties
Spark plasma sintering

ABSTRACT

Monolithic high purity CVD β -SiC materials were successfully joined with a pre-sintered Ti_3SiC_2 foil via solid-state diffusion bonding. The initial bending strength of the joints (~ 220 MPa) did not deteriorate at 1000°C in vacuum, and the joints retained $\sim 68\%$ of their initial strength at 1200°C . Damage accumulation in the interlayer and some plastic deformation of the large Ti_3SiC_2 grains were found after testing. The activation energy of the creep deformation in the temperature range of $1000 - 1200^\circ\text{C}$ in vacuum was ~ 521 kJmol⁻¹. During the creep, the linkage of a significant number of microcracks to form a major crack was observed in the interlayer. The Ti_3SiC_2 interlayer did not decompose up to 1300°C in vacuum. A mild and well-localized decomposition of Ti_3SiC_2 to TiC_x was found on the top surface of the interlayer after the bending test at 1400°C in vacuum, while the inner part remained intact.

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1. Introduction

Due to their excellent combination of properties, SiC monolithic and composite materials are primarily used as materials for aerospace and nuclear applications. In addition, SiC based composites are also being developed for aerospace and ground-based engine and hot structure fusion reactor applications [1]. In the nuclear field the most important application of SiC is for cladding materials in pressurised water reactors and flow channel insert materials in thermonuclear fusion reactors [2]. In fission power applications, fully ceramic SiC cladding and ceramic/metal cladding are two generic types of materials currently being considered for light-water reactors [3]. The fully ceramic cladding usually consists of a combination of fibrous SiC composites with a single layer or multiple layers of monolithic SiC coatings [3].

A Chemical Vapour Deposition (CVD) process can be used to produce high-purity, highly crystalline, fully dense (3.21 g/cm³) stoichiometric β -SiC [4,5]. CVD SiC has a cubic β -SiC structure

while hot-pressed or sintered SiC has the hexagonal α -SiC structure [5]. Among all SiC based materials, CVD β -SiC exhibits the highest elastic modulus and superior irradiation resistance [4]. CVD SiC is considered in aerospace and nuclear applications as a monolithic material or an external coating on SiC-matrix fibrous composites. In most cases, however, the ability of joining SiC based materials to themselves or other dissimilar materials is a critical technological need for their applications.

Among all of the joining approaches, only a few methods are appropriate for nuclear environments. It has been reported that diffusion bonding is a promising method of joining SiC for nuclear applications [3,6]. In recent years, Spark Plasma Sintering (SPS) has attracted considerable interest as a new advanced technique for the joining of both monolithic SiC [4,7–9] and ceramic matrix composites (CMCs) [10–12]. In this case, both a rapid heating and a short processing time allow a highly controllable reaction of the interlayer with the materials to be joined [4,8,10]. At the same time, the electric field can accelerate self-diffusion, and promote the migration of ions through the joining interface [8].

Apart from the use of the appropriate joining techniques, another critical issue is to develop new joining filler materials with high melting points, good oxidation and irradiation resistance. MAX

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phases (M is an early transition metal, A is an A-group element, and X is either C or N) are layered ternary materials with a unique combination of metallic and ceramic properties, such as damage tolerance, machinability, high temperature oxidation resistance, good electrical and thermal conductivities, thermal shock resistance, and wear resistance [13]. Moreover, they exhibit large plastic deformation at temperatures above 1200 °C [14]. Since MAX phases also possess excellent irradiation/corrosion resistance, they are regarded as promising structural materials for fusion reactors and lead-cooled fast reactors [15]. Among all of the MAX phase materials, Ti_3SiC_2 has been considered a promising candidate structural material for nuclear and high-temperature applications [8]. More recently, Ti_3SiC_2 has attracted considerable attention as a joining filler for SiC and CMC materials.

It is well known that Ti_3SiC_2 is the main phase formed when SiC is in a contact with Ti at high temperatures while TiSi_2 and TiC are secondary products [10,16]. This in-situ formation of the Ti_3SiC_2 in the joining interlayer resulted in a relatively high 4-point bending strength when SiC was joined with Ti (100 MPa [17], 126 MPa [4], and 250 MPa [18]). The formation of the Ti_3SiC_2 in the middle of the joined area was also observed when a Ti foil was used to join CMC materials [10]. While this reaction between Ti and SiC leads to an in-situ formation of a desirable Ti_3SiC_2 phase in the joining interlayer, rather high temperatures (1500 °C [18], 1700 °C [4,10]) were required to obtain the joints. In order to decrease the joining temperature, joining of SiC or CMCs with Ti_3SiC_2 as a joining filler rather than its in-situ formation has been considered. However, in most of the works the joining procedures relied on the reaction between the joining filler and the matrix to obtain a strong joint. Either a pre-synthesized Ti_3SiC_2 powder [7] or Ti_3SiC_2 tapes [8] were used to join monolithic SiC materials using hot-pressing (HP) and SPS technologies at temperatures from 1300 °C to 1600 °C. In both cases, the flexural strength of the joined samples increased with increasing joining temperature, reaching maximum values of ~ 110 MPa and ~ 99 MPa for HP and SPS joining processes, respectively. In both these works, decomposition of Ti_3SiC_2 occurred at temperatures ≥ 1400 °C, which was then followed by a chemical reaction with the SiC matrix. They concluded that the interface reactions were beneficial for obtaining sound joints as the lowest flexural strength was measured when no chemical reaction took place at 1300 °C. A similar beneficial influence of the decomposition of Ti_3SiC_2 followed by the reactions with the matrix on the shear strength of C_f/C composites was also reported [11]. In other words, although the Ti_3SiC_2 powders were used to join SiC and CMC materials, the best results were achieved when the amount of Ti_3SiC_2 decreased in the joining area due to both its partial decomposition and chemical reaction with SiC during joining.

On the contrary, in our recent work we demonstrated, for the first time, the advantages of the solid-state diffusion bonding over the solid-state reaction bonding when the pre-sintered Ti_3SiC_2 foil was used as a joining filler for the CVD-coated C_f/SiC and uncoated C_f/SiC , respectively [12]. In both cases, the sound joints were obtained using the SPS process at a temperature as low as 1300 °C, but the higher shear strength was measured when no reaction was found between the filler and the base material (~ 31 MPa vs. ~ 19 MPa for the coated and uncoated C_f/SiC composites, respectively).

Despite the fact that joining of SiC based ceramics has become an important issue for aerospace and nuclear applications (in both applications operating in extreme conditions); there is the lack of information on the mechanical performance and thermal stability of the joints at high temperatures. Very recently, Dong et al. reported on the high temperature bending strength of the SiC joints with the Ti_3SiC_2 powder joined at 1400 °C (up to 500 °C in air) and Ti/Si/C/Al powder joined at 1600 °C (up to 1200 °C in air) [9]. While in the first case the overall poor strength of the joints (66 MPa; due

to the porosity of the interlayer) significantly dropped to 21 MPa at 500 °C, in the second case the strength first decreased from 133 MPa to 68 MPa at 800 °C and then increased to 119 MPa at 1200 °C. However, the impurities in the Ti_3SiC_2 filler and their oxidation led to the formation of TiO_2 that significantly affected the strength results. On the other hand, Jiménez et al. performed thermal cycle tests on SiC- C_f/SiC joints with Ti_3SiC_2 (also containing TiC and TiSi_2) under simulated re-entry conditions with respect to the envisaged applications, i.e. temperature profiles under vacuum [19]. Up to 5 cycles at two testing temperatures (1391 °C and 1794 °C) showed no detectable damage; instead the amount of Ti_3SiC_2 increased after the test as TiC reacted with TiSi_2 . No other work on the high temperature stability and high temperature mechanical properties of the SiC based joints with Ti_3SiC_2 interlayer has been reported. Bulk Ti_3SiC_2 materials with different purities and microstructures were extensively investigated in the past and the high temperature mechanical properties of Ti_3SiC_2 are now well understood. However, it is not known whether its typical characteristics, such as brittle-ductile transformation at 1100 – 1200 °C, very high plasticity above 1200 °C, microcracks and cavity formation during deformation, etc., are retained when the Ti_3SiC_2 is constrained as the interlayer with a typical thickness up to ~ 100 μm between two ceramics. To the best of our knowledge, there has not been a reported systematic study on the high temperature stability and mechanical properties of monolithic CVD-SiC joints with high purity Ti_3SiC_2 interlayer (no unreacted TiC or TiC from decomposition of Ti_3SiC_2). The motivation for this work was to fill this gap and shed some light on the understanding of whether the Ti_3SiC_2 still keeps its characteristic of being a damage tolerant material with high plasticity even when confined as a layer in a SiC-SiC joint.

The aim of the present work was to develop a new joining technique for monolithic bulk CVD-SiC that would be appropriate for both aerospace and nuclear applications where the component geometry allows. Therefore, solid-state diffusion bonding at the temperature of 1300 °C was applied using SPS. Based on our previous investigation on joining of CMCs [12], a pre-sintered highly pure Ti_3SiC_2 foil was used as a joining filler to obtain a solid-state diffusion bonding. This is the first report on diffusion bonding of monolithic SiC materials when the Ti_3SiC_2 was used in the form of pre-sintered foils rather than powders [7,9] or tapes [8]. The main advantage of such a solid-state diffusion bonding is that neither densification nor reaction is required to produce joints. This avoids the possibility of undesirable shrinkage of the joining interlayer as well as the volume change upon in-situ production of the reaction products. The joining conditions using SPS technology were carefully chosen to avoid both the decomposition of Ti_3SiC_2 and the reaction between the joining filler and the high purity CVD-SiC matrix. The bending strength of the joined components was then investigated at room as well as high temperatures up to 1400 °C in vacuum. In addition, the creep resistance of the joints was studied in four point bending mode in the temperature range from 1000 °C to 1200 °C in vacuum. This work is the first study on the high temperature mechanical properties of the monolithic SiC materials joined with high purity Ti_3SiC_2 MAX phase (no TiC). This was done in order to determine the thermal stability of the joints in vacuum, thereby finding a potential temperature limit for the application of MAX phase interlayer as a joining filler.

2. Experimental procedure

2.1. Starting materials

The starting material was the high-purity (99.9995 %), fully dense (3.21 g/cm³) CVD β -SiC (The Dow Chemical Company). The

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