



# Combustion joining of carbon/carbon composites by a reactive mixture of titanium and mechanically activated nickel/aluminum powders

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Received 21 December 2012; received in revised form 27 February 2013; accepted 28 February 2013

## Abstract

Combustion joining of carbon/carbon (C/C) composites using a mixture of titanium and mechanically activated Ni/Al powders as a reactive medium is reported. A minimum preheating of the sample stack to 630 K is required to initiate the joining process. A robust crack- and pore-free joint layer ( $\sim 75 - 100 \mu\text{m}$  in thickness), which is composed of  $\text{NiAl}_x$  and  $\text{TiC}_y(\text{O}_z)$  phases, is produced. Tensile-strength testing of the joined C/C composites shows that the fracture does not occur along the joint layer.

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**Keywords:** A. Milling; A. Joining; B. Composites; D. Carbide

## 1. Introduction

Carbon/carbon (C/C) composites, due to their low density, high strength-to-weight ratio, as well as high thermo-elastic stability at elevated temperatures, are attractive thermo-structural materials for a variety of applications. These key characteristics enable use of C/C composites for manufacturing of rocket nozzles, noses and leading edges of reentry vehicles, as well as gas turbine engine components [1,2]. In addition, the high thermal-shock and wear resistance that C/C composites possess make them suitable for aircraft brake disks, where temperatures during harsh landing conditions (i.e., rejected take-off) can be as high as 2000 K [3–5].

As the demand for such materials increases, development of techniques for joining C/C composites with various complex structures is drawing more attention. Specifically, rapid and energy-efficient techniques for joining C/C composites are of great interest for industries such

as aircraft brake manufacturers. For example, one of the many possible applications is the refurbishment of carbon-brake disks by bonding a new piece to a used C/C core to produce a carbon-brake disk that meets special performance requirements. However, joining of refractory C/C composites is a challenging task. Mechanical or adhesive means could be used to join such materials but the applications are limited. For example, since carbon-brakes should be highly refractory components, C/C parts joined by traditional mechanical or adhesive joining methods would not hold up to harsh environments. Moreover, unlike metals, C/C composites do not lend themselves to welding, and even brazing can be difficult because many commonly used filler metals exhibit little or no wetting of carbon materials.

Few studies have reported joining of C/C composites. In general, the techniques that could be applied to join C/C composites include: reactive metal brazing [6], solid-state diffusion bonding [7,8] a combination of these approaches [9], as well as adhesive organic resin bonding [10] and hot-pressing [11]. As mentioned above, C/C composites joined via reactive metal brazing and adhesive bonding can be

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utilized only at relatively low temperatures. The other techniques require relatively long-time (hours), high-temperature (up to 2000 K) sample treatment and application of high external bonding loads. Such conditions often lead to the decrease of the materials' properties. Hence, a method for joining of C/C composites which is rapid and energy-efficient and can produce a robust refractory joint without a significant change of the properties of the materials to be joined is still a challenging and demanding task.

Combustion joining (CJ) is an attractive method for joining of a variety of materials [12,13]. It was derived from self-propagating high temperature synthesis (SHS), or combustion synthesis (CS), which was initially developed for materials synthesis. CJ is drawing attention as an effective tool for joining of refractory substances [14–18]. Some characteristics of CJ include: (i) short (seconds) processing time; (ii) energy efficiency, since the chemical energy of the internal system is primarily used for the production of the material; (iii) simple technological equipment; (iv) the ability to produce joint layers with functionally graded properties. White et al. [15] reported joining of refractory C/C composites via a class of refractory materials (carbides, borides, etc.) by CJ. However, a short-term ( $\sim 30$  s) but high-temperature (up to 2000 K) preheating of the joint stack was required in order to initiate the chemical reaction in the joint layer, which affected the composite properties.

Here we report an improved combustion-based technique for bonding of C/C composites by utilizing a CJ approach with a reactive mixture of titanium and mechanically activated Ni/Al powders. Incorporation of mechanically activated powders allowed initiation of the stack joining reaction after only preheating up to 630 K. The influence of the CJ parameters, including temperature, applied pressure and durations on the quality of the joint layer was investigated. The microstructure, phase and elemental composition of the joint layer were also studied. Finally, tensile-strength testing of joined C/C composites was performed.

## 2. Material and methods

### 2.1. Mechanical activation

The mechanical activation (MA) of Ni+Al mixture is described in detail elsewhere [19]. Short-term (15 min) high-energy ball milling (HEBM) of an equiatomic Ni+Al powder mixture (15 g of Ni+Al mixture per batch) was carried out using a PM100 (Retsch, Germany) planetary ball mill with a 250 mL stainless steel jar and 2 mm diameter stainless-steel balls as the milling medium in an inert atmosphere (argon). The ball-to-mixture ratio was 2:1. The rotational speed of the mill was 650 rpm. X-ray diffraction (XRD) analysis was performed to determine the phase composition of the initial Ni+Al mixture and after 15 min of MA. Results suggested that no new phases

(NiAl, NiAl<sub>x</sub> solid-solution, etc.) were formed after 15 min ball milling of Ni+Al powder mixture under the investigated conditions. It was shown [20] that such a mechanical activation treatment allows one to significantly decrease the self-ignition temperature of Ni/Al reactive mixture.

### 2.2. Materials preparation

Cylindrical C/C samples (10 mm in diameter  $\times$  12.5 mm in length) were fabricated from a commercial C/C aircraft brake disk (Honeywell Aerospace, South Bend, IN) with a density of  $1.67 \pm 0.04$  g/cm<sup>3</sup> and a total open porosity of  $\sim 15\%$ . A thin disk (0.3 g, 10 mm in dia.  $\times$  2 mm in height), cold-pressed from the powder medium, was placed in between the two C/C composite pieces to be joined. The disk was composed of 0.1 g of pure Ti powder ( $-325$  mesh, Alfa Aesar) and 0.2 g of mechanically activated Ni/Al composite with a molar ratio of 1:1 (initial Ni and Al powders,  $-325$  mesh, Alfa Aesar).

### 2.3. Joining method

A detailed description of the joining device and the experimental conditions is published elsewhere [21]. The stack (C/C–(Ti+Ni/Al+Ti)–C/C) to be joined was placed between two cooper electrodes in a specially designed press-die, which was connected to a DC power supply. The electrodes are part of a pneumatic system, which applies a uniaxial load to the sample. All operational parameters such as pressure ( $P$ ), applied current ( $I$ ), and others were defined by a programmable logic controller (PLC). As DC current passed through the stack, it was preheated due to the electrical resistance of the material. The experimental parameters used were initial load,  $P_i=4$  MPa; final load,  $P_f=20-40$  MPa; delay time between ignition and final load application,  $\Delta t=5$  msec; applied current,  $I=300-400$  A; joining duration,  $t=5$  s.

A high-speed infrared thermal imaging system (SC6000; FLIR Systems, Boston, MA) was used to monitor the temperature–time evolution of the process. Thermal images and videos were captured in frame sizes ranging from  $64 \times 8$  to  $640 \times 512$  over several different temperature ranges using commercial software (FLIR ThermaCAM Researcher).

### 2.4. Materials characterization

Cross-sections of joined C/C composites were prepared and microstructures, as well as elemental and phase compositions of the joint layers were examined with a scanning electron microscope (LEO series EVO 50), which was equipped with an Oxford Instrument EDS analyzer. X-ray diffraction was also performed (Scintag, X1 Advanced Diffraction System, Scintag Inc., USA). Joined C/C samples were machined to the desired configuration and tensile-strength tests ( $T_0=300$  K) were performed with a universal testing machine (Enduratec ELF 3000, Bose

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