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Current-Activated, Pressure-Assisted Infiltration: A Novel, Versatile Route for Producing Interpenetrating Ceramic–Metal Composites

Liangfa Hu^a, Ankush Kothalkar^a, Morgan O’Neil^b, Ibrahim Karaman^{a,b} and Miladin Radovic^{a,b*}

^aDepartment of Materials Science and Engineering, Texas A&M University, College Station, TX 77843, USA;

^bDepartment of Mechanical Engineering, Texas A&M University, College Station, TX 77843, USA

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A novel technique is proposed and demonstrated for processing interpenetrating ceramic–metal composites by the infiltration of molten metals/alloys into ceramic foams utilizing a current-activated, pressure-assisted technique. Ti₂AlC foams were prepared and used as the infiltration preform. Aluminum alloy 6061 (AA6061) has been infiltrated into the Ti₂AlC foams, resulting in AA6061/Ti₂AlC composites. The microstructure, composition, and distribution of phases in the composites were studied. The AA6061/Ti₂AlC composites were studied under compression testing, and the results were compared with those of the pure components of the composites. Advantages of this method in comparison to other methods for processing metal–ceramic composites are discussed.

Keywords: MAX Phase, Infiltration, Ceramic–Metal Composites, Aluminum Composites

1. Introduction Ceramic–metal composites have been under investigation for more than 40 years. On the one hand, adding a ceramic phase in light metals such as aluminum and magnesium results in composites with higher strength, elastic moduli, hardness, wear resistance and reduced coefficient of thermal expansion.[1] On the other hand, metal phases are incorporated in ceramics mainly to increase their toughness.[2] Among various methods for producing the ceramic–metal composites, such as powder metallurgy, stir casting, diffusion bonding, and deposition,[3] the infiltration of molten metals into ceramic preforms containing continuous open porosity is one of the most popular methods.[4] The processes for infiltration can be divided into two categories: (i) pressureless infiltration,[5] where the molten metals or alloys wet the ceramic preforms sufficiently to be spontaneously drawn into the preform, and (ii) pressure infiltration,[6] in which an external pressure is used to actively drive the molten metals or alloys into the preform.

Poor wettability of ceramic substrate with molten metal makes the pressureless infiltration process very slow or even in some instances impossible.[7] In addition, poor wetting can also yield weak bonding between the metal and ceramic phases, resulting in inferior

mechanical properties of the composites.[8] Therefore, numerous methods have been used to enhance the wettability. These methods include modifying the ceramic surface,[9] tailoring the chemistry of the metals and alloys,[9,10] the use of controlled atmospheres [10,11] and much higher temperatures than the melting points of the metals/alloys.[12] However, the components of the composites may be degraded due to chemical attack or introduction of new reaction phases at high temperatures.[13] In fact, the majority of infiltration-based composites are made by pressure infiltration processes. The external pressure can be mechanical as in squeeze infiltration,[14] or an applied gas pressure.[15] With the apparatus being made of steel, the squeeze infiltration is normally carried out at temperatures lower than the melting point of steel, while the gas pressure infiltration process normally requires a highly sealed container.[16] Moreover, both methods take relatively long time and may not be practical in many cases where the molten metals are highly reactive with the ceramic preforms.

Our interest concerns developing a rapid infiltration method based on a current-activated, pressure-assisted technique to minimize the two-phase reactions. The current-activated and pressure-assisted powder

*Corresponding author. Email: mradovic@tamu.edu

densification method utilizes simultaneously very large electric currents and applied loads to achieve rapid consolidation of ceramic or metallic powders.[17] In fact, it has been clearly shown that this method is viable to co-sinter highly reactive ceramic–metal systems.[18]

Aluminum (Al) and their alloys have been used extensively in various industries—such as automotive and aerospace—due to their lightweight, excellent corrosion resistance, good castability and machinability.[19,20] Two ternary carbides, namely Ti_2AlC and Ti_3AlC_2 , belonging to the family of MAX phases, where M is an early transition metal, A is an A-group element (from groups 13 to 16), and X is C and/or N, have high stiffness, high strength, good resistance to thermal shock and oxidation, and high thermal and electrical conductivities; they are also lightweight and easily machinable.[21,22] Such unique properties make them promising candidate materials for use as reinforcements in Al and its alloys and extend the application temperatures of Al and its alloys.[23] As a matter of fact, Ti_3AlC_2 /Al composites have been fabricated using powder mixture co-sintering.[23] The yield stress of the Ti_3AlC_2 /Al composite has been found to be twice as high as that of the Al matrix in the 20–500°C temperature range.[23] However, the Ti_3AlC_2 particles were barely sintered in the Ti_3AlC_2 /Al composites prepared by powder mixture co-sintering, because the co-sintering temperature was kept low to avoid substantial and harmful reactions. Therefore, to process interpenetrating Al/MAX phase composites with marginal two-phase reactions infiltration of molten metals/alloys into ceramic foams has to be considered. This method has been already used to process the Ti_2AlC /Mg composite that exhibits higher strength in tension and compression than other Mg composites, as well as high damping capacity.[24] However, pressureless infiltration is not an easy task because of the poor wettability of MAX phase preform with molten Al. The challenge for processing interpenetrating Al/MAX phase composites is therefore to achieve high density while controlling the reaction between MAX phase and Al.

The present paper reports on the current-activated, pressure-assisted infiltration (CAPAI) method for producing interpenetrating ceramic–metal composites where poor wettability and severe reactions prohibit application of conventional processing methods. Ti_2AlC foams were infiltrated by molten aluminum alloy 6061 (AA6061), resulting in AA6061/ Ti_2AlC composites. The composite microstructures were studied. The mechanical properties of the AA6061/ Ti_2AlC composites were characterized under compression testing, and the results were compared with those of the pure components of the composites, namely solution heat treated AA6061 and Ti_2AlC foam.

2. Experimental Details Ti_2AlC powders (MAXthal 211, Sandvik Heating Technology AB, Sörfvarnsvägen,

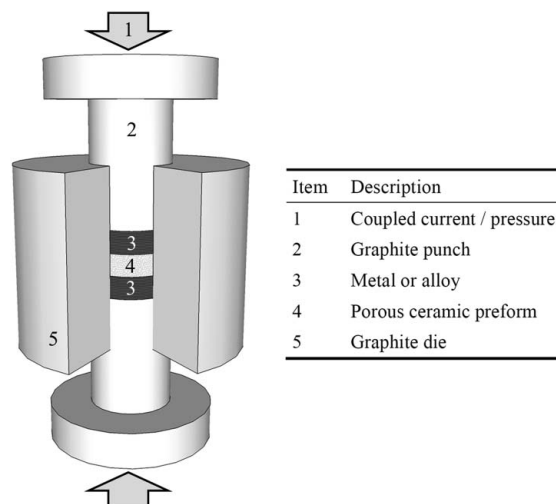


Figure 1. Schematic of the melt infiltration approach showing the coupled current/pressure (item 1), the graphite die set (items 2 and 5), and the ‘sandwich’-like set-up of the metal/alloy discs (item 3) and the porous preform disc (item 4).

Sweden) with the particle size in the 45–90 μm range, and NaCl powders (Sigma-Aldrich, St. Louis, MO, USA) with the particle size in the 355–500 μm range were used to process porous preforms following procedures described elsewhere in more detail.[25] The preparation of the Ti_2AlC foams involves three steps: (i) cold pressing a NaCl– Ti_2AlC powder mixture in 40/60 volume ratio; (ii) dissolving the NaCl in distilled water, and (iii) pressureless sintering of the porous green body under flowing argon at 1400°C for 4 h.

AA6061 discs (McMaster-Carr, GA, USA) with a diameter of 20 mm and a thickness of 4 mm were used for the infiltration process. As shown in Figure 1, a disc (20 mm in diameter and 4 mm in thickness) of Ti_2AlC foam was ‘sandwiched’ in between two AA6061 discs, and placed in a graphite die, followed by infiltration (SPS 25-10, GT Advanced Technologies, CA) at 750°C under 5 MPa uniaxial pressure for 1 min. A high dc current was applied, increasing from 0 to 1250 A in 4 min and then stabilized at 860 A for 1 min at the sintering temperature of 750°C. The pulse cycle was 10 ms on and 10 ms off. The ‘sandwich’ set-up enables more uniform infiltration of molten AA6061. The chamber was evacuated and held at 10^{-6} torr for 10 min before heating. The heating rate was 200°C per minute. It takes less than 10 min to process the samples, including heating, soaking, and cooling. Graphite foils were applied between the samples and graphite die before infiltration. The temperature was measured at the bottom graphite punch, which was immediately below the surface that was in contact with the sample.

The porosity of all samples was determined by the alcohol immersion method based on Archimedes’ principle, as specified in ASTM C20-00.[26] The theoretical

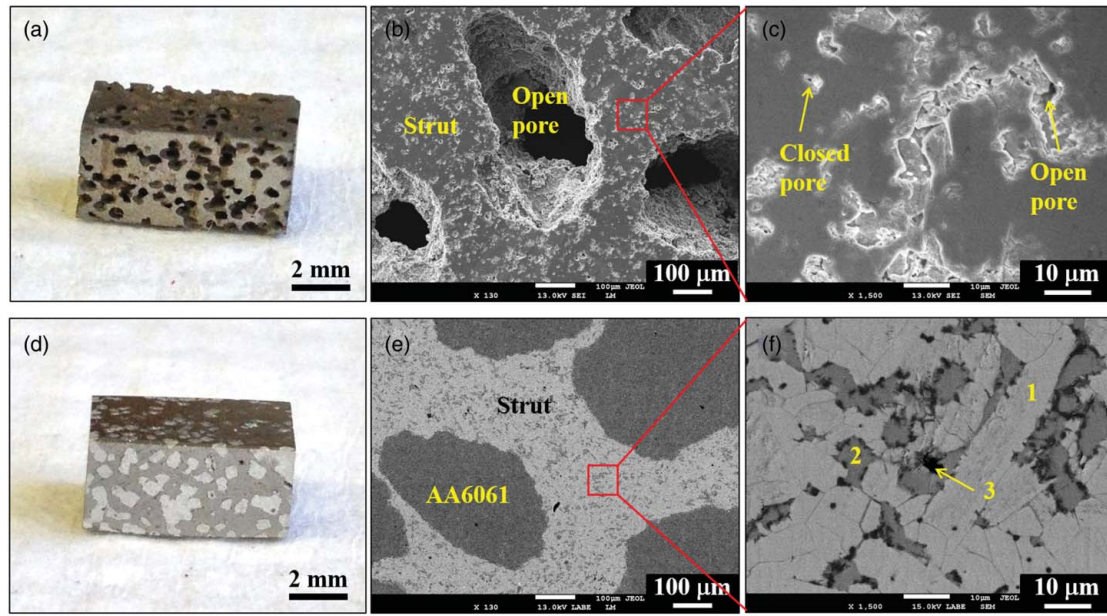


Figure 2. (a) A photograph of a porous Ti_2AlC preform, and SEM images of (b) the preform showing open pores and struts, (c) enlarged view on the strut showing both closed pores and open pores. (d) A photograph of an as-infiltrated AA6061/ Ti_2AlC composite, and SEM images of (e) the composite showing AA6061 (dark gray) infiltrated into the open pores, (f) enlarged view on the strut showing Ti_2AlC (1, light), titanium aluminide (2, gray), and Al_2O_3 (3, dark). The composition of phases 1, 2, and 3 are according to the EDS results (not shown here).

density values of $4.11 \text{ (g/cm}^3\text{)}$ [27] and $2.70 \text{ (g/cm}^3\text{)}$ [28] for fully dense Ti_2AlC and AA6061, respectively, were used to calculate the theoretical density of composites using the rule of mixture and assuming that the effect of the eventual reaction phases on the theoretical density is negligible. The relative density is thus the measured density value divided by the corresponding theoretical rule-of-mixture density value, i.e. 3.55 g/cm^3 .

The phase composition of starting powders and as-infiltrated composites was determined using an X-ray diffractometer, XRD (D8 Discover, Bruker, WI, USA) with $\text{Cu K}\alpha$ radiation (wavelength = $1.542 \text{ \AA}$) at 40 kV and 30 mA. The two theta range varied from 8° to 80° with a step size of 0.04° and a step time of 1.5 s. The XRD results were analyzed utilizing the Inorganic Crystal Structure Database (ICSD). The microstructure, phase composition, and distribution were characterized using a field emission scanning electron microscopy, SEM (JSM-7500F, JEOL, Tokyo, Japan) equipped with energy-dispersive spectroscopy (EDS). The accelerating voltage and emission current were 15 kV and 20 mA, respectively. The duration of spot scan of EDS was 60 s per spectrum. The compressive strength of the samples with dimensions of $3.5 \text{ mm} \times 3.5 \text{ mm} \times 7 \text{ mm}$ was measured by using a universal testing machine (MTS810, MTS, MN, USA) with a strain rate of $1.4 \times 10^{-4} \text{ s}^{-1}$. All samples for compressive testing were cut by electrical discharge machining.

3. Results and Discussions

3.1. Microstructure, Density, and Phase Analysis.

Figure 2(a)–(c) shows the Ti_2AlC foam prepared with 40 vol.% NaCl pore former with particle size in the $355\text{--}500 \text{ }\mu\text{m}$ range. The open and closed porosity of the preform are $35.7 \pm 1.0 \text{ vol.}\%$ and $5.1 \pm 0.4 \text{ vol.}\%$, respectively, which means that 87.5% of overall porosity (sum of the open and closed porosity) are open. Both SEM and optical images in Figure 2(a)–(c) show that the open pores are homogeneously dispersed in the Ti_2AlC , while some closed pores can be found in the struts of the foam due to the incomplete sintering of Ti_2AlC particles during the pressureless sintering. Meanwhile, Figure 2(c) clearly illustrates the strong connectivity of Ti_2AlC grains on the struts of the foam. The strong network of grains is critical to obtain high mechanical strength of porous preform, which turns into the struts of the composite after infiltration. As a component of the infiltrated composite, the strong Ti_2AlC networks play an important role to reach high compressive strengths in the composites.

For infiltration processes, ceramic preforms have to be made before the metal is infiltrated. The preform structures are normally packed particles or fibers, [29] and they can in some instances be solid foams. [30] The mechanical behavior of the preform is important, not only during handling and placement of the preform, but also because the preform may be stressed during the pressure infiltration. MAX phases are kinking-nonlinear elastic solids which can dissipate significant amount of energy during cyclic

loading due to the formation and annihilation of fully reversible incipient kink bands.[31] Previous investigations have shown exceptional damage tolerance of MAX phase foams.[25] Specifically, the compressive strength of Ti_2AlC decreases almost linearly with increasing porosity and less rapidly when compared with common ceramics, such as Al_2O_3 , Si_3N_4 , and ZrO_2 , whose compressive strength drops dramatically with increasing porosity.[32] In addition to relatively high strength of Ti_2AlC foams, another advantage of using them as preforms is the easy control of pore size and porosity which allows producing interpenetrating ceramic–metal composites with customized structures.

Figure 2(d)–(f) shows a photograph and the microstructure of the AA6061/ Ti_2AlC composite processed by the CAPAI method. The open and closed porosity are 1.0 ± 0.9 vol.% and 4.6 ± 0.2 vol.%, respectively. More than 97% of all open pores are successfully infiltrated with AA6061 in the composites. Although the open porosity dramatically decreased due to infiltration with AA6061, the closed porosity hardly changed after infiltration. Therefore, the majority of the residual porosity in the composites comes from closed pores that are of micrometer size and isolated in the Ti_2AlC area. In contrast, pores or voids can hardly be found in the AA6061 areas and in the two-phase boundaries. This suggests that the AA6061 infiltration occurred dominantly in the open pores, and volume fractions of the components in the final composites, e.g. ceramic phase, metallic phase, open and closed pores, are tunable by adjusting the initial porosities in the ceramic preform. Note that the amount of reaction phase in the AA6061/ Ti_2AlC composite processed in this work is much smaller than that in the previously reported $\text{Ti}_3\text{AlC}_2/\text{Al}$ composites prepared by powder mixture co-sintering.[23] Indeed, Ti–Al–C phase diagram [33] clearly shows that Al reacts with Ti_2AlC at 727°C to form stable phases like titanium aluminide intermetallic phases. Even when the $\text{Ti}_3\text{AlC}_2/\text{Al}$ composites were processed at a lower temperature by the co-sintering method, that method takes much longer time than the infiltration process and thus results in more reaction between two phases. This suggests that the CAPAI method effectively reduces the reactions in highly reactive ceramic–metal systems.

Figure 3 shows the XRD results of the starting materials, AA6061 and Ti_2AlC foam, and the as-infiltrated AA6061/ Ti_2AlC composites. The results clearly show that the Ti_2AlC foam contains, in addition to Ti_2AlC , some Ti_3AlC_2 and a very small amount of Al_2O_3 . These two impurity phases come from the starting commercial MAXtal211 powder. Ti_2AlC , Ti_3AlC_2 and Al are three major phases in the AA6061/ Ti_2AlC composite in the presence of a small amount of Al_2O_3 . All phases identified in the composites were also found in the starting materials, namely AA6061 and Ti_2AlC foam, and no new phases were detected by the XRD. Although a new

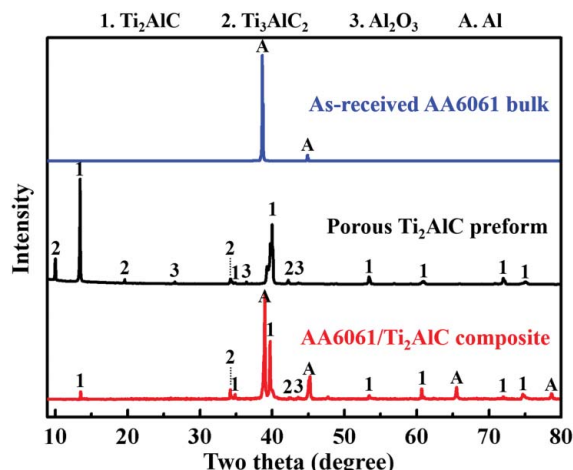


Figure 3. XRD results of the AA6061/ Ti_2AlC composite and its pure components, namely AA6061 and Ti_2AlC foam. All XRD data were collected at room temperature. The identification of phases was done according to the ICSD collection code 165460 for Ti_2AlC , 153266 for Ti_3AlC_2 , 10425 for Al_2O_3 , 43423 for Al.

phase, intermetallic titanium aluminide (Figure 2(f)), was identified by the EDS in the AA6061/ Ti_2AlC composite, its amount is so small that they are not detectable in the XRD.

The CAPAI technique is utilized here for the first time for the infiltration process to take advantage of its three inherent benefits of CAPAI. First, the coupled current/pressure provides simultaneous heating and pressing that actively drive the molten metals into the preform. Second, the fast and controllable heating rate, as high as $500^\circ\text{C}/\text{min}$, offers an easy and efficient way to avoid the reactions between the ceramic preforms and the metal/alloy melts during infiltration. Third, the controllable atmosphere, i.e. either vacuum or inert gas, makes it easy to investigate the effects of atmosphere on the infiltration mechanism and to control possible oxidation and reactions between ceramic and metallic phases.

Particular advantages of this technique arise when one compares the CAPAI method to conventional infiltration methods. First, unlike the time-consuming pressureless infiltration processes, the CAPAI process can be very fast and efficient, e.g. 10 min for a single infiltration process, including heating/melting, infiltrating and cooling/solidification. Second, in contrast to the pressure infiltration where temperature is limited below the melting point of steel and high cost is associated with sealing, the infiltration temperature for the CAPAI can be as high as 2200°C and sealing for highly pressurized gas is not required. Note that the current or electric field could not be excluded as a factor of enhancing the wettability of 6061 Al onto the MAX phase, even though the mechanism is not clear at this point.

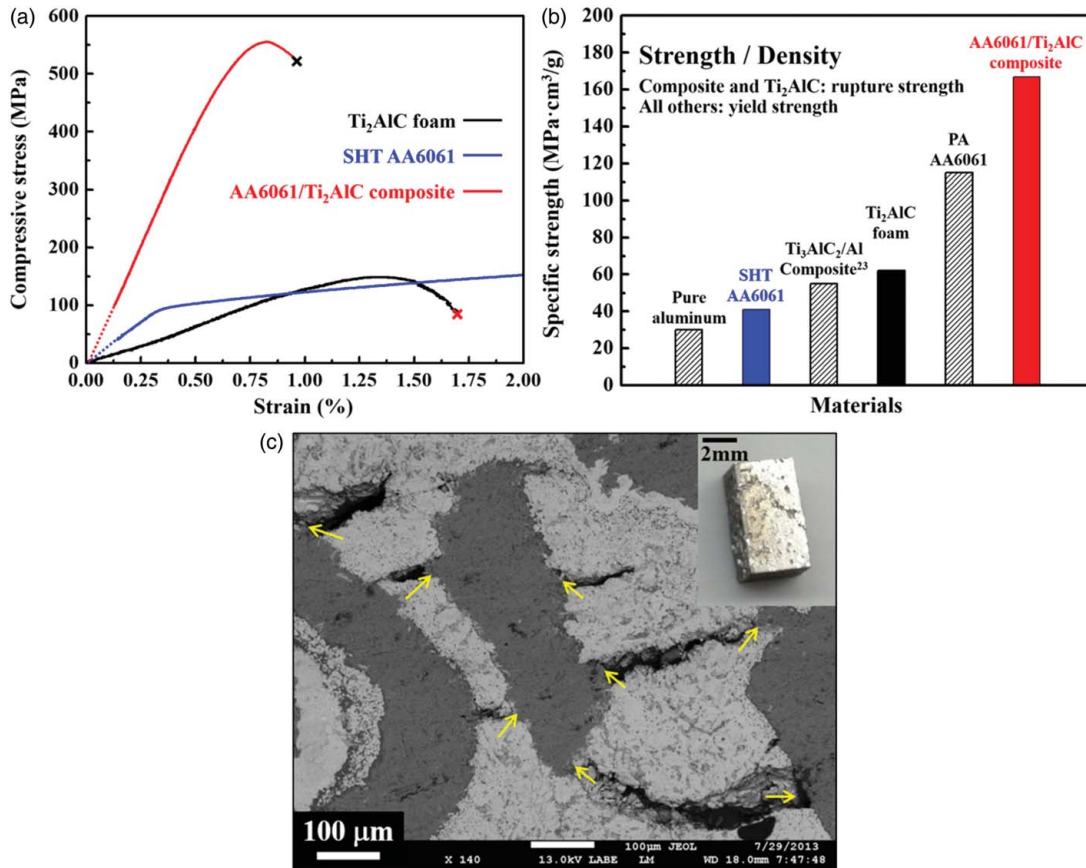


Figure 4. (a) Compressive stress–strain curves of the AA6061/ Ti_2AlC composites, SHT AA6061, and Ti_2AlC foam. (b) Specific strength of the AA6061/ Ti_2AlC composites, peak aged (PA) AA6061, and Ti_2AlC foam, the $\text{Ti}_3\text{AlC}_2/\text{Al}$ composites,[23] SHT AA6061, and pure aluminum. (c) Surface morphology of the post-compression composite. The inset shows that the sample failed by cracking in the direction of approximately 45° relative to the loading direction with a tortuous crack path. The cracks propagate from bottom right to top left. The arrow indicates that cracks in the ceramic phase were effectively stopped and deflected by metallic phase.

3.2. Mechanical Properties. It has been demonstrated up to this point that the CAPAI method allows processing novel ceramic–metal composites which could not otherwise be obtained using powder metallurgy and conventional infiltration techniques. Furthermore, the section below also demonstrates that the CAPAI method can also result in superior properties in the composites when compared to their pure components.

Figure 4(a) shows the compressive stress–strain curves of the AA6061/ Ti_2AlC composites, solution heat treated (SHT) AA6061 (to be more comparable with its condition in the composite), and Ti_2AlC foam. The compressive strength of the AA6061/ Ti_2AlC composite is 560 MPa, which is about four times as high as that of the Ti_2AlC foam (150 MPa) and about five times as high as the yield strength of the SHT AA6061 (110 MPa [34]). More importantly, Figure 4(b) shows that the specific strength, i.e. compressive/yield strength divided by density, of the AA6061/ Ti_2AlC composite is 167 $\text{MPa cm}^3/\text{g}$, which is about 3 times as high as that of the Ti_2AlC foam (62 $\text{MPa cm}^3/\text{g}$), 1.5 times as

high as that of the peak aged AA6061 (115 $\text{MPa cm}^3/\text{g}$ [34]), 3 times as high as that of previously reported value of the $\text{Ti}_3\text{AlC}_2/\text{Al}$ composite (55 $\text{MPa cm}^3/\text{g}$ [23]), and 4 times as high as the yield strength of the SHT AA6061 (41 $\text{MPa cm}^3/\text{g}$). Interestingly enough, post-testing analysis of fracture surfaces (Figure 4(c)) shows distinctive evidence of crack propagation resistance of the AA6061/ Ti_2AlC composite.

As the inset in Figure 4(c) shows, all tested samples failed by cracking in the direction of approximately 45° relative to the loading direction with a tortuous crack path. This observation suggests that the composite shows a graceful failure, and does not crush and fragment under compression. Higher magnification SEM image in Figure 4(c) of the area around the fracture surface clearly shows that cracks in the ceramic phase were effectively stopped and deflected by the metallic phase. More importantly, no cracks in the interfacial areas have been observed after compression testing, indicating exceptionally strong bonding between two phases in the composite.

4. Conclusions In summary, the CAPAI method is proposed and demonstrated as a novel, efficient and rapid technique for producing interpenetrating ceramic-metal composites by the infiltration of molten metals and alloys into porous ceramic preforms. AA6061/Ti₂AlC composites were successfully processed using this method. The major findings are summarized as follows:

- (1) More than 97% of the open porosity was infiltrated with molten metal even after a very short processing time. Most of the residual pores in the composites are closed pores and are found in the Ti₂AlC area as a result of incomplete sintering during preparation of the preform. This method offers an efficient route for producing interpenetrating ceramic-metal composites with customizable structures by controlling the structure of the ceramic preform.
- (2) Little reaction is observed in the AA6061/Ti₂AlC system, which suggests that the CAPAI method allows processing novel, highly reactive ceramic-metal composite systems which could not otherwise be obtained using powder metallurgy and conventional infiltration techniques.
- (3) This method can result in composites with superior properties: the specific compressive strength of the AA6061/Ti₂AlC composites is about 1.5 times of the specific yield strength of peak aged AA6061.

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