Mechanical Response of Fine Grained Ti₂AlC under Extreme Thermo-mechanical Loading Conditions

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Abstract

An experimental investigation was conducted to evaluate the compressive constitutive behavior and fracture initiation toughness of fine grained (~4.2 μm) Ti₂AlC in dynamic and quasi-static loading at different temperatures. A Split Hopkinson Pressure Bar (SHPB) apparatus was used in conjunction with an induction coil heating system for dynamic experiments at elevated temperatures, while a universal mechanical testing machine equipped with an extensometer and furnace was used for quasi-static compressive testing. A series of experiments were conducted at different temperatures from 25 °C to 1200 °C and strain rates of 10⁻⁴ s⁻¹ and 500 s⁻¹. A single edge notched specimen was used to determine the fracture initiation toughness in dynamic and quasi-static loading from ambient temperature to 900 °C. The SHPB apparatus was modified for this purpose and high speed photography was incorporated to determine fracture initiation time, and the load corresponding to this time was used to calculate the dynamic fracture toughness. The results of this study reveal that the peak compressive failure stress in dynamic conditions decreases with increasing temperature, from 1600 MPa at room temperature to 850 MPa at 1200 °C. In the dynamic testing condition, the failure remains predominately brittle even at temperatures as high as 1200 °C. Under static loading conditions, the peak compressive stresses were always lower than those in dynamic loading conditions, but they also decreased monotonically with increasing temperature. Also under static conditions, a significant drop in peak compressive failure stress and an increase in ductility was observed above the brittle-to-plastic transition temperature of around 900 °C. The fracture experiments reveal that the strength and fracture toughness in dynamic conditions are higher than the corresponding quasi-static values by approximately 35% at room temperature and that they both decrease with increasing temperatures. It was found that high temperature and low strain rate promote plastic behavior of Ti₂AlC.

Keywords: MAX Phases, SHPB, Fracture Toughness, Brittle to Plastic Transition, Kink Bands

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Introduction

As a member of the family of ternary carbides and nitrides generally referred to as MAX phases, Ti_2AlC has a unique combination of properties that makes it attractive for different applications. All MAX phases share general chemical formula of $\text{M}_{n+1}\text{AX}_n$ (where M is an early transition metal, A is a group element, and X is carbon and/or nitrogen), and distinctive nanolayered structure in which M_{n+1}X_n layers are interleaved with pure A-group element layers [1–3]. In addition, MAX phases display an unique combination of metal- and ceramic-like properties including high stiffness, good thermal and electrical conductivity, excellent thermal shock resistance, high damage tolerance, high fracture toughness and ease of machinability with conventional tooling [1–5]. Out of the more than 70 MAX phases discovered to date, Ti_2AlC is considered to be one of the most promising candidates for high temperature applications owing to its superior oxidation resistance due to formation of a self-healing Al_2O_3 protective layer that is well adhered and spallation resistant even during thermal cycling [6–11]. In addition, it has low density (4.11 g/cm^3) [3], high thermal ($46 \text{ W/(m}\cdot\text{K)}$) and electrical ($4.42 \times 10^6 \text{ S/m}$) [6] conductivities, and a high Young's modulus ($\approx 280 \text{ GPa}$) [12].

It is well established by now that the quasi-static mechanical response of all MAX phases studied to date changes from brittle-like at room temperatures to pseudo-plastic at elevated temperatures [2,5,13–15]. Although MAX phases are fundamentally brittle at near room temperature, their stress-strain response in quasi static cyclic loading is non-linear elastic and hysteretic, with all but the first stress-strain hysteresis loops being closed, reversible and reproducible [15–18]. At elevated temperatures, they all show brittle-to-plastic transition (BPT) usually at temperatures between $900 - 1100^\circ\text{C}$ [2,5]. Above the brittle-to-plastic transition temperature (BPTT), their strength decreases rapidly with temperature and their mechanical response becomes pseudo-ductile with strains to failure in both tension and compression exceeding 25 % [1,2,19–23]. In the case of polycrystalline Ti_3SiC_2 , the best characterized MAX phase to date, BPTT depends on the grain size and strain rate [13,14,21,24,25].

Fracture toughness of MAX phases have also been studied mainly in quasi-static loading conditions and been reported to vary substantially from 4 to $16 \text{ MPa m}^{1/2}$ [26–29]. The discrepancies among these reported fracture toughness values can likely be attributed to the different compositions, grain sizes, testing methods, and experimental conditions of the studies themselves. In addition, Ti_3SiC_2 (and most likely other MAX phases) exhibit substantial resistance curve (R-curve) behavior since the stress needed to extend a crack increases with crack length [27]. Although the number of papers reporting on high temperature fracture toughness of MAX phases is very limited [26,28,29], they all show that it drops significantly at about the BPTT. The latter suggests that activation of secondary slip systems recently proposed in the literature [15,30] almost certainly does not play a major role in pseudo-ductile behavior of MAX phases above BPTT, as it was discussed by elsewhere [2]. Our current understanding of the aforementioned mechanical behavior of MAX phases under quasi static loading is mostly based on the facts that they are highly plastically anisotropic as basal and only basal plane dislocations can easily move, multiply, and arrange either in pileups or in walls/arrays even at room temperature. In addition, MAX phases can also deform by kinking and kink band formation throughout the temperature ranges tested. At high temperature, delamination and formation of cavitation and microcracks have also been used to account for observed large strains [25,31].

Most of the literature has focused on characterization of mechanical behavior of MAX phases in general, and Ti_2AlC in particular, at the quasi-static strain rates [13,14, 19,32–35]. To the best of the authors' knowledge, only one high strain rate study [36] for MAX phases has been reported to date, but it was restricted to room temperature experiments and a very narrow range of strain rates. However, that study demonstrated that the Split Hopkinson Pressure Bar (SHPB) technique could be used to test mechanical behavior of Ti_2AlC in the 2600 – 4700 s^{-1} strain rate range and that no apparent strain rate dependencies can be observed for the strain rates tested. Analysis of fracture surfaces showed that kinking was an active deformation mechanism, even at the high strain rates tested. However, the commercially available samples used in that study contained large grains, with lengths exceeding 150 μm (see Figure 6 in [36]). Large grains are more prone to kinking, and the samples in that study [36] also contained significant amounts of impurities whose role in the overall mechanical response of Ti_2AlC under dynamic loads is still elusive. Therefore, since Ti_2AlC has been suggested to operate in extreme environments, there is a clear need to investigate the mechanical response of MAX phase under high temperatures and dynamic loadings, in order to further develop an understanding of the deformation mechanisms in this ceramic material. The objective of this study is to investigate the effect of temperature and loading rate on the constitutive behavior and fracture initiation toughness of Ti_2AlC . An experimental investigation of the constitutive behavior under quasi-static and dynamic loading is conducted at different temperatures ranging from 25°C to 1200 °C. A single edge notched specimen is used in a three point bend configuration to evaluate the fracture initiation toughness. The quasi-static and dynamic fracture initiation toughness is investigated at 25 °C, 500 °C and 900 °C. A modified SHPB apparatus is utilized to investigate the dynamic fracture initiation toughness and high speed photography is incorporated to determine fracture initiation time.

2. Materials and Experimental Procedure

2.1 Materials

Commercial Ti_2AlC powder (Maxthal 211, Sandvik Heating Technology, Sweden) was used to process all samples in this study. Bulk Ti_2AlC discs 50 mm in diameter were sintered using a spark plasma sintering system (SPS 25-10, GT Advanced Technologies, CA) in a graphite die at 1300 °C for 15 minutes under a constant load of 100 MPa. The relative density of the bulk Ti_2AlC was measured to be above 98% using the alcohol immersion method, based on Archimedes' principle [37,38]. All of the specimens employed in this study were electron-discharge machined from the 50 mm disks of bulk Ti_2AlC . X-ray diffraction (D8 Discover, Bruker, USA) results of the powder showed that it consisted of ~65 vol.% Ti_2AlC , ~25 vol.% Ti_3AlC_2 and 10 vol.% TiAl_x intermetallic impurities. Figure 1 shows the scanning electron microscopy (SEM) (Quanta 600 FEG, FEI, USA) micrograph in backscatter electron mode of an examined Ti_2AlC sample, and the TiAl_x impurities can be clearly observed as darker gray areas in the aforementioned image. Measurements carried out on over 100 grains in the SEM micrographs of the polished and etched samples (not shown here) showed that the average diameter and thickness of the MAX phases grains (Ti_2AlC or Ti_3AlC_2) were 4.2 μm and 2.1 μm , respectively. From the analysis of SEM images together with quantitative energy-dispersive spectroscopy (EDS) of undetached samples (not shown here), the amount of TiAl_x was determined to be around 7 vol.%.

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138 *2.2 Quasi-static characterization of constitutive behavior*

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140 Room and high-temperature quasi-static uniaxial compression testing of cylindrical
 141 samples (5 mm in diameter by 8 mm in length) was conducted using a servo-hydraulic testing
 142 machine (MTS-810, MTS, USA) equipped with a split tube furnace and SiC pushrods. Tests
 143 were performed under a constant crosshead displacement rate chosen to result in an initial quasi-
 144 static strain rate of 10^{-4} s^{-1} . Heating rates were limited to $600 \text{ }^{\circ}\text{C hr}^{-1}$ and a soaking time of 15
 145 minutes was used to ensure that uniform temperature was reached in the sample before testing. A
 146 high temperature axial extensometer (632.59, MTS, USA) equipped with SiC extension rods was
 147 directly attached to the spacers. True stress and true strains in the specimen were calculated as

$$148 \quad \sigma_s = \frac{FL_i}{A_0L_0} \quad \dots (1)$$

$$149 \quad \varepsilon_s = -\ln\left(\frac{L_i}{L_0}\right) \quad \dots(2)$$

150 where F is the instantaneous load, L_i is the instantaneous length of the specimen, A_0 is the
 151 original cross-sectional area of the specimen, and L_0 is the original length of the specimen.

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153 *2.3 Dynamic characterization of constitutive behavior*

154 A Split Hopkinson Pressure Bar (SHPB) was used to study the dynamic behavior of
 155 Ti_2AlC . The striker bar, incident bar and transmission bar in the SHPB setup were all made out
 156 of Maraging steel. The incident and transmission bars had a diameter of 12.5 mm and lengths of
 157 2133 mm and 1524 mm, respectively. The striker bar was propelled using an air-operated gun.
 158 The Ti_2AlC specimens had a diameter of 6.35 mm and a thickness of 3.18 mm. To account for
 159 the hard and brittle nature of Ti_2AlC , modifications were made to the SHPB setup following
 160 procedures used to test other ceramic materials [39]. A lead pulse shaper of 1 mm thickness was
 161 placed at the impact end of the incident bar as shown in Figure 2. Two tungsten-carbide (WC)
 162 inserts were placed between the two bars and the specimen was sandwiched between the inserts.
 163 The impedance of the inserts was matched to that of the bars, hence the inserts do not disturb the
 164 stress wave profiles. The inserts were used to reduce stress concentration in the specimens and to
 165 prevent indentation of the specimens into the bars. Molybdenum disulfide was used to lubricate
 166 the specimen insert interface to minimize the effects of friction. A high-speed digital camera
 167 (SA1, Photron, Japan) was used at a frame rate of 300,000 fps to capture the deformation
 168 process. The camera image capturing was triggered from the oscilloscope recording strain gauge
 169 data, resulting in synchronized strain and image measurements.

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171 For high temperature experiments an induction coil heater was used in conjunction with
 172 the SHPB setup. The Ti_2AlC specimens are electrically conductive, and therefore could be
 173 heated by electromagnetic induction using coiled loops around the specimen. The WC inserts
 174 prevented the development of a sharp temperature gradient in the bars and also protected the
 175 strain gauges mounted on the bars. The welding of thermocouples to the specimens was observed
 176 to produce minor cracks in them. To avoid this, a calibration procedure was developed to
 177 evaluate the amount of power needed in the induction heating system to achieve the desired

steady state temperature of the specimen in about 30 seconds. The calibration method is robust since temperature only changes by about $\pm 2\%$ as the specimen is displaced 5 mm from its central location within the induction coil. This time- and power-based calibration was used to heat the specimens during the high temperature experiments.

The measurements from the strain gauge on the incident bar had to be resolved into an incident wave with high magnitude and a reflected wave with low magnitude. Hence, to enable this recording, the output from both of the strain gauges on incident bar were recorded using two different channels, one channel having a high scale and one having a low scale. Using one-dimensional wave theory, the engineering stress and engineering strain in the specimen can be determined from the transmitted and reflected strain pulses, respectively, as

$$S_s = E_b \frac{A_b}{A_s} \varepsilon_t(t) \quad \dots (3)$$

$$e_s = \frac{-2c_b}{L_s} \int_0^t \varepsilon_r(t) dt \quad \dots (4)$$

where S_s and e_s are stress and strain in the specimen, ε_r and ε_t are the time resolved strain values of reflected and transmitted pulses, c_b is the longitudinal bar wave speed, E_b is the Young's modulus of the bar material, A_b is the cross-sectional area of the bar, and A_s is the cross-sectional area of the specimen and L_s is the thickness of the specimen. The true stress and true strain were calculated as:

$$\sigma_s = S_s(1 - e_s) \quad \dots (5)$$

$$\varepsilon_s = -\ln(1 - e_s) \quad \dots (6)$$

2.4 Quasi static and dynamic fracture initiation toughness

The specimens used for the fracture toughness (inset in Figure 3) experiments were fabricated by electron discharge machining using a 0.1 mm diameter. The quasi-static fracture initiation toughness was investigated using a three-point bending experiment at, 25 °C, 500 °C and 900 °C. The specimens were heated to the desired temperature using an induction heating system before applying the load. The experiments were conducted under a fixed loading rate of 1 mm/min at room temperature. A loading rate of 5 mm/min was used at elevated temperatures to provide the same rate of stress intensity factor increase and to avoid creep effects. The experiments showed a clear and well defined peak load at which the crack initiates. The static fracture initiation toughness, K_{IS} , was determined using this peak load as:

$$K_{IS} = \frac{F_{Peak}}{B\sqrt{W}} f\left(\frac{a}{W}\right) \quad \dots (7)$$

where F_{Peak} is the peak load, B the specimen thickness, W the specimen width, a the initial crack length and $f(a/W)$ is a geometric factor.

A modified SHPB apparatus with an induction heating system was used to investigate the dynamic fracture initiation toughness (Figure 3). The apparatus mainly consists of an incident bar, striker bar and pressure gun. The incident bar and striker bar are made from T6061 aluminum. The strains in the incident bar are measured using two semiconductor strain gauges that are attached in the middle of the bar diametrically opposite to one another. The backed semiconductor strain gauges (type, SS-090-060-1150 PB-S1; resistance, $1125 \pm 75 \text{ X}$; gauge factor 155 ± 10 ; manufactured by Micron Instruments) are about 75 times more sensitive than the foil type gauges and are preferred to capture small strain signals generated during dynamic fracture experiments. During loading, the specimen was sandwiched between the incident bar and the rigid frame. For an elevated temperature experiment, the bar was first kept apart and the specimen is heated to the desired temperature using the induction heater (usually about 50°C higher than the test temperature). The bar was later brought manually into contact with the specimen. The temperature of the specimen was monitored with a thermocouple, which was spot welded onto the specimen. Once the specimen is in contact with the incident bar, the striker bar is propelled towards the incident bar by an air-operated gun. The incident and reflected strain signals are recorded using a high frequency Vishay 2301A signal-conditioning amplifier that was connected to an oscilloscope. The load-time history at the specimen/bar interface is obtained from the recorded strain data using one dimensional elastic wave theory given by the following equation:

$$F(t) = [\varepsilon_i(t) + \varepsilon_r(t)]EA \quad (8)$$

where F is the force, ε_i and ε_r are the incident and reflected strain pulses, E is the Young's modulus, and A is the cross sectional area of the bar. When the time of fracture initiation is sufficiently long enough to permit several reverberations of the stress wave along the width of the specimen, the dynamic stress intensity factor, K_I , can be calculated from the input load as follows:

$$K_I(t) = \frac{F(t)}{B\sqrt{W}} f\left(\frac{a}{W}\right) \quad (9)$$

The dynamic fracture initiation toughness (K_{ID}) corresponds to the stress intensity factor at the time of crack initiation, i.e. $K_{ID} = K_I(t_{\text{initiation}})$.

3. Results and Discussion

3.1 Constitutive Behavior under dynamic and static loading

Typical time shifted segments of the recorded strain pulses that are used for the analysis of dynamic behavior are shown in Figure 4. The specimen undergoes strain rate acceleration for the first $20 \mu\text{s}$ as seen by the rising reflected pulse. After $20 \mu\text{s}$, and up to the time of failure, the specimen experiences an average strain rate of 550 s^{-1} . The catastrophic failure of the specimen begins at $38 \mu\text{s}$. The real time images captured using the high speed camera are displayed in Fig. 5. The time of occurrence of these images is indicated on the transmitted wave profile in Figure 4

with a box (■) and corresponding image number. Frame 1 in Figure 5 shows the condition at the beginning of the experiment, whereas frame 2 shows the compressed state of the specimen, due to relative motion of the WC inserts, right before the onset of peak stress. At 38.8 μs (frame 3 in Figure 5), the first surface cracks appeared, which would have initiated at the peak stress state, along with several internal cracks that could not be observed at that moment. The multiple cracks that can be observed after 42 μs (frame 4 in Figure 5) resulted in a dramatic decrease of load bearing capacity (Figure 4), and complete fracture of the specimen soon after that.

The force ratio during a typical experiment, defined as the ratio of the forces exerted on the two end faces of the specimen, is plotted with respect to time in Figure 6. It can be seen from this figure that the dynamic stress equilibrium (a force ratio of 1) was attained at about 10 μs and was maintained up to the point of specimen failure. The stress equilibrium condition is necessary for the validity of the SHPB results and needs to be ascertained in each experiment.

The true stress-strain curves of Ti_2AlC in compression during quasi-static and dynamic loadings at various temperatures are plotted in Figure 7. During dynamic loading, the stress in the specimens increased with the strain until it reached a critical stress, at which the specimens failed catastrophically. The peak compressive stress varies between 1427 MPa and 1870 MPa, for the room temperature experiments in dynamic loading conditions. Those values are significantly higher than peak stresses of 517-907 MPa reported earlier by Bhattacharya et al. [36] for Ti_2AlC tested under dynamic loadings. The higher peak stresses reported here cannot be attributed to strain-rate effect, since Bhattacharya et al. [36] tested Ti_2AlC samples at much higher strain rates ranging between 2600 and 4700 s^{-1} . Even in quasi static condition, the compressive strength of 1263 MPa reported here at room temperature is also the highest ever reported for Ti_2AlC [1,2,35,40–46]. The high values of peak stresses in both quasi-static and dynamic conditions reported here can be attributed mostly to the microstructural effects, i.e to the Hall-Petch effect because the grain size of samples used in this study was much finer than that used in previous works , and possibly they had larger amount of impurities such as TiAl_x that are quite common in Ti_2AlC as a result of incomplete reaction sintering [47]. It is also worth noting that, in general, the variations in peak stress were narrower for high temperature experiments (Figure 8) and in quasi-static loading conditions. The large spread in the dynamic failure stress at room temperature could be due to two reasons: (a) the strain rate in these experiments is close to the critical stain rate beyond which fracture mechanics fully governs the failure process resulting in large failure stress. This critical strain rate might be lower at higher temperatures; (b) there could be also some larger variation in the microstructure or pre-existing defects from sample to sample.

The peak compressive stresses from Figure 7 are plotted as a function of temperature in Figure 8 for both dynamic and quasi-static loading conditions. Figure 8 clearly shows that the compressive strength values are higher under dynamic loading conditions than in quasi-static loading, at all temperatures. Despite the fact that the room temperature compressive strength value of 1263 MPa, measured in this study, is the highest ever reported for Ti_2AlC at room temperature, it decreases rapidly above 800-900 $^{\circ}\text{C}$ and reaches a value of 175 MPa at 1150 $^{\circ}\text{C}$. The peak compressive stress in the high strain rate experiments also decreases monotonically with increasing temperatures from 1645 MPa at room temperature to 1315 MPa at 1000 $^{\circ}\text{C}$. Above that temperature the peak compressive stress measured in dynamic conditions decreases

rapidly with increasing temperature, reaching the value of 853 MPa at 1200 °C. It is also worth noting here, that the variations in peak stress were narrower for high temperature experiments.

Figure 7 also clearly shows that under quasi-static loading, Ti₂AlC exhibits near brittle behavior to fracture up to 700 °C. Starting at 900 °C, the sample displays elastic and large pseudo-plastic deformation indicating the onset of a BPT around that temperature. It is important to note that the samples tested at 1000°C and 1100°C under quasi-static loading did not fail, but the test had to be stopped after reaching strains of ~26% due to the limit in the measurement range of the extensometer. Under dynamic conditions, strains to failure are significantly lower when compared to quasi-static values, and very small plastic strains (< ~0.5%) following peak loads can be observed only in samples tested above 1000 °C. For dynamic experiments conducted at an average strain rate of 500 s⁻¹, all specimens exhibited catastrophic failure at all testing temperatures, i.e. they shattered in a large number of small pieces. The inset in Figure 7 shows typical specimen recovered at the end of a room temperature dynamic experiment. Even at 1200 °C, the specimen shattered after testing showing mainly brittle failure at high strain rate, regardless of the relatively high failure strains of 1.5 % . Note that horizontal axis in Figure 7 has an axis break to represent the large strains observed in these samples which exhibited large plastic deformations and barreling in quasi-static loading conditions.

Overall, the compressive strength decreases with increasing temperature, and this decrease is more pronounced above the BPTT in both quasi-static and dynamic conditions. More importantly, Figure 8 clearly indicates that BPT occurs at much lower temperature, i.e. at around 800-900 °C in quasi-static loading. The same transition at high strain rates can be observed in the 1000-1100 °C temperature range. Similar BPT was first observed in Ti₃SiC₂ MAX phase [20], and later in many other MAX phases [15,19,22,28] including Ti₂AlC [32] to occur near 1000°C for in quasi static loading conditions. Radovic et al. [13,21] demonstrated for the first time that this BPT temperature in Ti₃SiC₂ is grain size dependent and more importantly strain rate dependent, shifting to higher temperatures for higher strain rates. Results in Figure 8 further support that finding, as BPTT in dynamic loading is higher for ~200-300 °C, than in quasi-static conditions. Also, the fact that strength decreases monotonically in a similar manner in both static and dynamic conditions below BPT, suggests lower strain rate sensitivity in this temperature range. Above BPTT, the stress decreases more significantly with temperature in quasi-static conditions, suggesting high strain rate sensitivity in the mechanical response of Ti₂AlC. Strain rate sensitivity (*m*) can be determined at different strains and temperatures from the true stress – true strain curves, similar to those in Figure 7, as:

$$m = \left. \frac{\partial \ln \sigma}{\partial \ln \dot{\epsilon}} \right|_{\epsilon, T} \approx \left. \frac{\ln(\sigma_1 / \sigma_2)}{\ln(\dot{\epsilon}_1 / \dot{\epsilon}_2)} \right|_{\epsilon, T} \quad \dots(10)$$

where $\dot{\epsilon}$ and σ are strain rate and stress, respectively. Table 1 summarizes strain rate sensitivity values at different strains and temperatures. The values of *m* in Table 1 show very low strain rate sensitivity below BPTT, ranging from 0.061-0.077, that increase gradually to values of 0.091-0.155 above BPTT. Even above BPTT, strain rate sensitivity values reported here for Ti₂AlC are still lower than the values of 0.42-0.52 reported for Ti₃SiC₂ [21], or 0.23-0.70 for Ti₃AlC₂ [15], for the reasons that are not clear at this point. However, the lower values of strain rate sensitivity reported here might be related to the fact that samples examined here had very fine grain size and that loading rates varied for more than six orders of magnitude, unlike in previous studies.

Table 1. Strain rate sensitivity (m) at different strains and temperatures:

Temperature, °C	Strain Rate Sensitivity (m) at strains of		
	0.2%	0.3%	0.4%
25	0.065	0.077	0.073
500	0.072	0.067	0.061
1000	0.091	0.109	0.114
1100	0.153	0.155	0.155

3.2. Fracture Toughness

A typical load-time plot obtained during a dynamic fracture experiment is shown in Figure 9. The vertical red line in the plot indicates the time at which crack initiates, and the load corresponding to this point was subsequently used to calculate the dynamic fracture initiation toughness using equation 9. One of the challenges in the dynamic fracture initiation toughness experiment is to find a method to accurately determine the crack initiation time. Researchers have used strain gauges [48–50], graphite gauges [51,52] and silver paint circuits [53] to identify the time of fracture initiation for room temperature and high temperature experiments up to 800 °C. These techniques are difficult to implement in high temperature experiments for conductive materials. Hence, in the present study, high speed photography was incorporated to identify the time corresponding to the load required to initiate the crack. A Photron SA1 high-speed digital camera was used at a frame rate of 450,000 fps with an image resolution of 128 x 32 pixels. The specimen was coated with a white colored high temperature ceramic paint to enhance the contrast of the crack with respect to the specimen. The camera was synchronized to ensure both that the images and strain gauge data could be correlated and that the total time from the beginning of the load to the fracture initiation could be evaluated. This enabled the determination of the total time history from the beginning of the load to the fracture initiation.

This time of fracture initiation was ascertained using the synchronized high speed images of the crack tip area. Fig. 10 shows typical high speed images used to capture crack initiation. The crack was seen to grow in the 37th frame that was taken 80 μ s after the application of load on the specimen.

The quasi-static and dynamic fracture initiation toughness of Ti_2AlC as a function of temperature and loading rate are shown in Figure 11, and decrease with an increase in temperature. The quasi-static fracture initiation toughness at room temperature (25 °C) is measured to be 6.8 $\text{MPa m}^{1/2}$, but decreases to 3.73 $\text{MPa m}^{1/2}$ at 900 °C. For evaluating dynamic fracture initiation toughness as a function of temperature for Ti_2AlC , two experiments were conducted at each temperature and the error bars in Figure 11 indicate the range of values obtained. As seen in the Figure 11, the dynamic fracture initiation toughness is 9.105 ± 0.021 $\text{MPa m}^{1/2}$ at room temperature, and is 8.615 ± 0.064 $\text{MPa m}^{1/2}$ at 900 °C. The rate of change of stress intensity, $\dot{K}$ was on the order of 10^4 $\text{MPa m}^{1/2} \text{s}^{-1}$ for the dynamic experiments and 1 $\text{MPa m}^{1/2} \text{s}^{-1}$ for the quasi-static experiments. Thus the fracture initiation toughness is also loading rate dependent for this material, showing higher values of fracture toughness under dynamic conditions. Similar observations have been previously reported for many other brittle ceramics such as Al_2O_3 , SiC , Si_3N_4 [54–56]. For all temperatures, fracture toughness is higher under dynamic loading as compared to static loading. Furthermore, the decrease in fracture toughness

with temperature is more drastic under quasi-static loading. During the dynamic fracture toughness experiments the fracture process zone does not have enough time to grow due to inertia effects. Therefore, the specimens bear larger loads before the crack initiates. These loads translate into higher toughness values under dynamic loading. This can also explain observed differences in compressive strengths between samples tested in dynamic and static conditions below BPTT temperatures, since preexisting defects would propagate at much lower stresses at lower strain rates. Additional confirmation of this conclusion can be found in the fact that both fracture toughness and peak compressive stress are higher by approximately the same amount (35%) at room temperature. This is expected for brittle solids in which fracture mechanics fully govern the failure process and in which the strength can be defined as $\sigma = K_I / \sqrt{2\pi a}$. Unfortunately, because of limitations of experimental setup, we were not able to measure fracture initiation toughness at temperatures above BPTT in this study, especially in dynamic loading conditions.

The microscopic images of fracture surfaces from the fracture toughness experiments are shown in Figure 12. Images (a) and (b) depict the specimen fracture surface after quasi-static loadings at room temperature and 900 °C, respectively. It is well established by now that monolithic polycrystalline MAX phases owe their relatively high fracture toughness and good damage tolerance to kinking and kink band formation that can be promoted by increasing the grain size [1,2,27]. As shown in images (a) and (c), the grain boundaries are rather clear and there are some instances of kinking. However, as expected for very fine grained microstructure examined here, the amount of kinking is much smaller than previously observed by Bhattacharya et al. [36] on the fracture surfaces of coarse grained Ti₂AlC tested in dynamic conditions. Although, there is no evident difference between fracture surfaces in quasi-static and dynamic loading at room temperature (Figures 12 a and c), they show different morphologies after testing at 900 °C. Fracture surfaces of samples tested in dynamic conditions at 900 °C, i.e. below BPTT for dynamic loading conditions, still show some evidence of kinking as the energy dissipation mechanisms (Figure 12 d). However, grains deformed by kinking can hardly be observed on the fracture surfaces after testing in quasi-static conditions at the BPTT of 900 °C for quasi-static testing conditions. Instead, the larger amount of delaminated grains can be observed (see Figure 11b). The latter is in very good agreement with results in Figure 11, which show more moderate decrease in fracture initiation toughness with temperature in dynamic conditions, as kinking and kink bridging [1,2] are operative at all temperatures in those conditions. However, the fact that very little kinking but significant grain delamination and intergranular cracks can be observed on the fracture surfaces after testing in quasi-static loading at temperature close to BPTT, suggest that delamination and fragmentation of individual grains is much easier than kinking, leading to the more pronounced decrease in fracture initiation toughness. Last but not least, it is worth noting that results shown here are the first evidence of kinking in any of the MAX phases with such a fine grain size of 4.2 mm diameter and 2.1 μm thickness.

6. Summary

An experimental investigation was conducted to evaluate the constitutive behavior and fracture initiation toughness of Ti₂AlC under thermo-mechanical loading. For both quasi-static and dynamic loadings, Ti₂AlC exhibits brittle failure for room temperature experiments. The peak compressive stress for failure decreased with increasing temperatures for both quasi-static as well as dynamic loadings, and this decrease is more pronounced above so called brittle-to-

ductile transition temperature (BPTT) in both cases. However, BPTT was higher in dynamic loading condition than in quasi-static conditions for 200-300 °C. In quasi-static loading conditions, BPTT was observed in the 800-900 °C temperature range and, above that temperature, significant plastic strains exceeding 25% were measured. In dynamic conditions, that BPTT was observed in 1000-1100 °C, and even at 1200 °C the samples failed in brittle manner with very little plasticity (strain to failure <1.5%). Ti₂AlC shows positive strain rate sensitivity exhibiting higher peak compressive stress with increasing strain rate.

The fracture toughness was also found to be loading rate sensitive: it showed an increase with the rate of loading from 6.8 MPa m^{1/2} in quasi-static to 8.615± 0.064 MPa m^{1/2} in dynamic loading conditions at room temperature. The fracture toughness was seen to decrease with increasing temperature for both quasi-static and dynamic loadings, although this decrease was more significant at quasi-static loading condition. The SEM images indicate significant kink band formation in fractured specimens, but their number is significantly lower on the fracture surfaces of samples tested in quasi-static loading conditions at higher temperatures.

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