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Characterization of Novel Materials Under Extreme Dynamic Loading Conditions

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CHARACTERIZATION OF NOVEL MATERIALS UNDER EXTREME DYNAMIC
LOADING CONDITIONS

BY

PRATHMESH NAIK PARRIKAR

A DISSERTATION SUBMITTED IN PARTIAL FULFILLMENT OF THE
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IN

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DOCTOR OF PHILOSOPHY DISSERTATION

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ABSTRACT

To improve the performance, efficiency and safety of future equipment's in commercial, aerospace, nuclear and defense structures, worldwide efforts are being directed towards the development of novel materials which exhibit superior structural and multifunctional capabilities in extreme environments. Fundamental investigation into the thermo-mechanical response and dynamic failure of the materials is paramount before they can be incorporated into the design of future space access vehicles that can operate reliably in combined, extreme environments. For this purpose, a comprehensive study was conducted to evaluate the performance of variety of materials such as Ti_2AlC , Ti_3AlC_2 , Hastelloy X and 2024 Aluminum under extreme thermo-mechanical loadings.

An experimental investigation was conducted to evaluate the compressive constitutive behavior and fracture initiation toughness of fine grained ($\sim 4.2 \mu\text{m}$) Ti_2AlC 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. 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 calculate the dynamic fracture toughness. The results from these experiments reveal that the peak compressive failure stress in dynamic conditions decreases with increases in 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 . However, brittle-to-plastic transition was observed at around 900°C under quasi static loadings. The fracture experiments reveal that the dynamic fracture toughness is higher than the quasi-static value by

approximately 35%. The effect of grain size on the constitutive behavior of Ti_2AlC from room temperature to 1100°C under dynamic and quasi-static loading was also investigated using high density Ti_2AlC samples of three different grain sizes. The results show that under quasi-static loading the specimens experience a brittle failure for temperatures below Brittle to Plastic Transition Temperature (BPTT) of $900\text{--}1000^\circ\text{C}$ and pseudo-plastic behavior at temperatures above the BPTT. During dynamic experiments, the specimens exhibited brittle failure, with the failure transitioning from catastrophic failure at lower temperatures to graceful failure at higher temperatures, and with the propensity for graceful failure increasing with increasing grain size. The compressive strengths of different grain sizes at a given temperature are related to the grain length by a Hall-Petch type relation.

Experimental studies were then conducted to investigation of mechanical response and fracture toughness of Ti_3AlC_2 under dynamic loading at different temperatures. SHPB apparatus in conjunction with an induction coil heater were used to dynamically load the specimen at various temperatures. The results of this study reveal that Ti_3AlC_2 exhibits brittle failure during dynamic loading, even for temperatures of 1100°C . The peak compressive stress for failure decreased with increasing temperatures. The fracture toughness was also seen to decrease with increasing temperature. The SEM images indicate that under dynamic loading conditions the deformation behavior and fracture mechanisms do not change with temperature.

The constitutive behavior of Hastelloy X was evaluated over a wide range of strain rates. Shear compression specimen (SCS) geometry was utilized to achieve strain rates from 10^{-3} - 20000 s^{-1} . A screw driven testing machine and a SHPB apparatus were used to load the specimens. An induction coil heating system was utilized in conjunction with SHPB to evaluate constitutive behavior at elevated temperatures under dynamic loading. Room temperature experiments were carried out at varying strain rates from 10^{-3} - 20000 s^{-1} exhibit

the strain rate sensitivity of Hastelloy X. The rate sensitivity increases after rate exceeds 1000 s^{-1} . The stress strain curves at various temperatures under dynamic loading showcase a yield strength anomaly. The stress peaks at the beginning of dynamic yielding at 850°C and 1000°C .

An experimental investigation was conducted to understand the effect of curvature on blast response of aluminum panels under extreme temperatures. Three aluminum 2024-T3 panels: flat panel and two curved panels, with radii of curvatures of 304.8 mm and 111.8 mm were used for the study. A shock tube apparatus was utilized to impart controlled shock loading. Propane flame torches were used to heat the specimens and experiments were conducted at 25°C , 200°C and 350°C . High speed photography coupled with 3D Digital Image Correlation (DIC) technique was used to obtain full field deflection and velocity, as well as in-plane strain on the back face of the panels. The different modes of deformation and the mode transitions during the blast loading of the three panels have been identified. The effect of temperature on the deformation modes is investigated. The deflections of panel with 304.8 mm radius of curvature were found to be higher than the other two panels. With increase in temperature, the deflections increased and strain was seen to get localized.

Experiments were performed to study the shear banding phenomenon in Ti6Al4V. SCS of Ti6Al4V were used in this of study the formation and evolution of adiabatic shear bands. The gage section of specimen was speckled using high temperature paint and SHPB apparatus will be used to dynamically load the SCS of Ti6Al4V. High speed photography was used to capture the deformation of speckled specimen. The high speed images were analyzed using DIC software to extract the strain fields during shear banding. Before dynamic failure localization in strain field were noticed. These localizations indicate towards formation of shear bands.

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PREFACE

Experimental studies have been conducted to study the thermomechanical response of materials under extreme environments. Fundamental investigation into the thermo-mechanical response and dynamic failure of the materials is paramount for the design of future commercial, aerospace, nuclear and defense structures that can operate reliably in combined, extreme environments. This dissertation addresses the dynamic behaviors and the failure mechanisms of aerospace materials under extreme thermo-mechanical loadings. This dissertation is prepared using the manuscript format.

Chapter 1 focuses on an experimental investigation to evaluate the compressive constitutive behavior and fracture initiation toughness of fine grained ($\sim 4.2 \mu\text{m}$) Ti_2AlC 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 the dynamic fracture toughness. The results 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. Under static loading conditions, the peak compressive stresses were always lower than those in dynamic loading conditions. 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. This chapter follows the formatting guidelines specified by Materials Science and Engineering: A.

Chapter 2 details the study conducted to investigate effect of grain size on the constitutive behavior of Ti_2AlC from room temperature to 1100°C under dynamic and quasi-static loading. High density Ti_2AlC samples of three different grain sizes were studied. A series of experiments were conducted at different temperatures from 25°C to 1100°C for strain rates of 10^{-4} s^{-1} and 400 s^{-1} . The results show that under quasi-static loading the specimens experience a brittle failure for temperatures below Brittle to Plastic Transition Temperature (BPTT) of $900\text{-}1000^\circ\text{C}$ and pseudo-plastic behavior at temperatures above the BPTT. During dynamic experiments, the specimens exhibited brittle failure, with the failure transitioning from catastrophic failure at lower temperatures to graceful failure at higher temperatures, and with the propensity for graceful failure increasing with increasing grain size. The compressive strengths of different grain sizes at a given temperature are related to the grain length by a Hall-Petch type relation. This chapter follows the formatting guidelines specified by Mechanics of Materials.

Chapter 3 details the experimental investigation conducted to evaluate the mechanical response of Ti_3AlC_2 under dynamic loading at different temperatures. A SHPB apparatus in conjunction with an induction coil heater was used to perform dynamic compression experiments. A series of experiments were conducted at temperatures from 25°C to 1200°C at an average strain rate of 500 s^{-1} . Edge notched bend specimens were used to determine the dynamic fracture initiation toughness from ambient temperature to 900°C with a modified SHPB setup. High speed photography was incorporated to determine fracture initiation time during the dynamic fracture toughness experiments. The results of this study reveal that

Ti₃AlC₂ exhibits brittle failure during dynamic loading, even for temperatures of 1200 °C. The peak compressive stress for failure decreased with increasing temperatures. The fracture toughness was also seen to decrease with increasing temperature. The SEM images indicate that under dynamic loading conditions the deformation behavior and fracture mechanisms do not change with temperature. This chapter follows the formatting guidelines specified by Experimental Mechanics.

Chapter 4 provides the details of experimental investigation conducted to study the constitutive behavior of Hastelloy X over a wide range of strain rates. Shear compression specimen (SCS) geometry was utilized to achieve strain rates from 10⁻³-20000 s⁻¹. A screw driven testing machine and a SHPB apparatus were used to load the specimens. An induction coil heating system was utilized in conjunction with SHPB to evaluate constitutive behavior at elevated temperatures under dynamic loading. Room temperature experiments were carried out at varying strain rates from 10⁻³-20000 s⁻¹ exhibit the strain rate sensitivity of Hastelloy X. The rate sensitivity increases after rate exceeds 1000 s⁻¹. The stress strain curves at various temperatures under dynamic loading showcase a yield strength anomaly. The stress peaks at the beginning of dynamic yielding at 850 °C and 1000 °C. This chapter follows the formatting guidelines specified by Experimental Mechanics.

Chapter 5 details the experimental study to understand the effect of curvature on blast response of aluminum panels under extreme temperatures. Three aluminum 2024-T3 panels: flat panel and two curved panels, with radii of curvatures of 304.8 mm and 111.8 mm were used for the study. A shock tube apparatus was utilized to impart controlled shock loading. Propane flame torches were used to heat the specimens and experiments were conducted at 25 °C, 200 °C and 350 °C. High speed photography coupled with 3D Digital Image Correlation (DIC) technique was used to obtain full field deflection and velocity, as well as in-plane strain on the back face of the panels. The different modes of deformation and the mode transitions

during the blast loading of the three panels have been identified. The effect of temperature on the deformation modes is investigated. The deflections of panel with 304.8 mm radius of curvature were found to be higher than the other two panels. With increase in temperature, the deflections increased and strain was seen to get localized. This chapter follows the formatting guidelines specified by International Journal of Impact Engineering.

Appendix A describes the experiments were performed to study the shear banding phenomenon in Ti6Al4V. SCS of Ti6Al4V were used to observe the formation and evolution of adiabatic shear bands. The specimen dynamically loaded by using the SHPB apparatus. High speed photography was used to capture the deformation of speckled specimen. DIC technique was used to evaluate the strain fields during shear banding. Before dynamic failure localization in strain field were noticed. These localizations indicate towards formation of shear bands.

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Chapter 1

Mechanical Response of Fine Grained Ti_2AlC under Extreme Thermo-mechanical Loading Conditions

by

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Abstract

An experimental investigation was conducted to evaluate the compressive constitutive behavior and fracture initiation toughness of fine grained ($\sim 4.2 \mu\text{m}$) Ti_2AlC 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 Ti_2AlC .

Keywords

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

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 $2600 - 4700 \text{ s}^{-1}$ 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 $150 \text{ }\mu\text{m}$ (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.

Materials and Experimental Procedure

Materials

Figure 1: Backscatter electron micrograph of etched Ti_2AlC where dark gray phase identified by EDS to be TiAl_x .

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.%.

Quasi-static characterization of constitutive behavior

Room and high-temperature quasi-static uniaxial compression testing of cylindrical samples (5 mm in diameter by 8 mm in length) was conducted using a servo-hydraulic testing machine (MTS-810, MTS, USA) equipped with a split tube furnace and SiC pushrods. Tests were performed under a constant crosshead displacement rate chosen to result in an initial quasi-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 minutes was used to ensure that uniform temperature was reached in the sample before testing. A high temperature axial extensometer (632.59, MTS, USA) equipped with SiC extension rods was directly attached to the spacers. True stress and true strains in the specimen were calculated as

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

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

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

Dynamic characterization of constitutive behavior

A Split Hopkinson Pressure Bar (SHPB) was used to study the dynamic behavior of Ti_2AlC . The striker bar, incident bar and transmission bar in the SHPB setup were all made out of

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

Figure 2: Schematic representation of SHPB setup

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

developed to 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:

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

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

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.

Figure 3: Schematic representation of modified SHPB setup for dynamic fracture toughness experiments. Inset shows the dimensions of edge notched specimen

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. $K_{ID} = K_I(t_{\text{initiation}})$.

Results and Discussion

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 μs as seen by the rising reflected pulse. After 20 μs , 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 μs . 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.

Figure 4: Typical strain pulses recorded during the SHPB experiment

Figure 5: Real time images during SHPB experiment

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.

Figure 6: Typical force equilibrium conditions at the specimen-bar interface

Figure 7: True compressive stress-strain curve of Ti_2AlC at various temperatures in quasi static (strain rate of 10^{-4} s^{-1}) and dynamic (strain rate of 500 s^{-1}). Inset of typical post-mortem specimen after a room temperature dynamic 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 strain 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 °C and reaches a value of 175 MPa at 1150 °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 °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 8: Effect of temperature and loading rate on compressive strength. Error bars show standard deviation from 3 or more tests.

Figure 7 also clearly shows that under quasi-static loading, Ti_2AlC 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:

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

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 BPPT, 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 (<i>m</i>) 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

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.

Figure 9: Typical Load–Time plot for dynamic loading at 500 °C

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.

Figure 10: Typical high speed images during for dynamic loading at 500 °C

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 $9.105 \pm 0.021 \text{ MPa m}^{1/2}$ at room temperature, and is $8.615 \pm 0.064 \text{ MPa m}^{1/2}$ 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.

Figure 11: Effect of temperature on the fracture initiation toughness of Ti_2AlC under quasi-static and dynamic loading.

Figure 12 (a) and (b) depict the fracture surface after quasi-static loadings at 25 °C and 900 °C, respectively; (c) and (d) depict the fracture surface after dynamic loadings at 25 °C and 900 °C, respectively. Red circles in (b) mark some delaminated grains.

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 Ti_2AlC 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.

Summary

An experimental investigation was conducted to evaluate the constitutive behavior and fracture initiation toughness of Ti_2AlC under thermo-mechanical loading. For both quasi-static and dynamic loadings, Ti_2AlC 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%). Ti_2AlC 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 $6.8 \text{ MPa m}^{1/2}$ in quasi-static to $8.615 \pm 0.064 \text{ MPa m}^{1/2}$ 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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Chapter 2

The effect of grain size on deformation and failure of Ti_2AlC MAX Phase under Thermo-mechanical loading

by

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Abstract

The effect of grain size on the constitutive behavior of Ti_2AlC from room temperature to 1100°C under dynamic and quasi-static loading was investigated. High density Ti_2AlC samples of three different grain sizes were densified using Spark Plasma Sintering and Pressureless sintering, respectively. A Split Hopkinson Pressure Bar (SHPB) apparatus in conjunction with an induction coil heating system was used for dynamic experiments at elevated temperatures and a servo-hydraulic testing machine equipped with vertical split furnace and SiC pushrods was used for quasi-static compressive testing. A series of experiments were conducted at different temperatures from 25°C to 1100°C for strain rates of 10^{-4} s^{-1} and 400 s^{-1} . The results show that under quasi-static loading the specimens experience a brittle failure for temperatures below Brittle to Plastic Transition Temperature (BPTT) of $900\text{--}1000^\circ\text{C}$ and pseudo-plastic behavior at temperatures above the BPTT. During dynamic experiments, the specimens exhibited brittle failure, with the failure transitioning from catastrophic failure at lower temperatures to graceful failure at higher temperatures, and with the propensity for graceful failure increasing with increasing grain size. The compressive strengths of different grain sizes at a given temperature are related to the grain length by a Hall-Petch type relation.

Introduction

Ti_2AlC belongs to a family of ternary carbides and nitrides called MAX phases. MAX phases, with the general chemical formula $\text{M}_{n+1}\text{AX}_n$, are a class of nanolayered, machinable, ternary compounds, where M is an early transition metal, A is a group 13–16 element, X is a carbon and/or nitrogen, and M_{n+1}X_n layers are interleaved with pure A-group element layers (Barsoum, 2000; Barsoum, 2013; Barsoum and Radovic, 2011). MAX phases exhibit a unique combination of properties, including ceramic-like properties such as high temperature strength, high elastic modulus, and excellent corrosion/oxidation resistance, and metallic-like

properties such as good thermal and electrical conductivity, good machinability, thermal shock resistance, and damage tolerance (Barsoum, 2000; Barsoum, 2013; Barsoum and El-Raghy, 1996; Barsoum and Radovic, 2011; Radovic and Barsoum, 2013; Sun, 2011). Ti_2AlC has superior oxidation resistance which is attributed to formation of a well adhered self-healing Al_2O_3 protective layer (Basu et al., 2011; Byeon et al., 2007; Sundberg et al., 2004; Tallman et al., 2013; Wang and Zhou, 2003; Yang et al., 2011) and has low density (4.11 g/cm^3) (Barsoum, 2000) making it a prospective candidate for a number of high temperature applications.

The studies to date on the quasi-static behavior of all MAX phases report that mechanical response changes from brittle-like at room temperatures to pseudo-plastic at elevated temperatures from $900 - 1100^\circ\text{C}$ (Barsoum, 2013; Barsoum and Radovic, 2011; Radovic et al., 2002; Radovic and Barsoum, 2013; Zhang et al., 2015; Zhen et al., 2005). MAX phases' 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 (Barsoum et al., 2003; Benitez et al., 2016; Shamma et al., 2015; Zhou and Barsoum, 2010). It is well established by now that dislocation plays an important role in the mechanical behavior of Ti_2AlC and other MAX phases as they are numerous but confined in the basal plane. Basal plane dislocations can also easily move, multiply, and arrange either in pileups or in walls/arrays, even at room temperature. This makes plastic deformation of MAX phases grains highly anisotropic (Barsoum et al., 1999; Farber, 1999; Farber et al., 1998; Guitten et al., 2014). Under applied stress, this plastic anisotropy leads to large internal stresses in the grains unfavorably oriented for deformation by dislocation glide in polycrystalline MAX phase; favorably oriented grains deform easily by basal dislocation glide. In addition to basal plane slip, MAX phase can also deform by kinking, kink band formations, delaminations, formation of microcracks and cavities, and grain boundary sliding (Barsoum, 2013; Barsoum and

Radovic, 2011, 2004; Poon et al., 2011; Radovic and Barsoum, 2013). Several studies have been conducted to see the effect of grain size on the mechanical properties of Ti_3SiC_2 (El-Raghy et al., 1999; Radovic et al., 2002; Zhen et al., 2005) in quasi static loading conditions, as different deformation mechanisms depends on the grain size in different ways. All these studies indicate that fine grained materials exhibit higher compressive strength compared to coarse grained materials, but at the same time lead to the more brittle and less graceful failure of the MAX phases, with smaller hysteresis loops (Benitez, 2015).

There have been only a few high strain rate studies [28–30] on MAX phases that have been reported to date. Bhattacharya et al. (2014) conducted experiments on coarse grained Ti_2AlC containing a large amount of secondary phases (mostly TiAl_x intermetallic) in the 2600 – 4700 s^{-1} strain rate range, and showed that kinking was an active deformation mechanism, even at the high strain rates. Naik Parrikar et al. (2016) studied fine grained Ti_2AlC under quasi static and dynamic conditions and showed that in the dynamic testing condition, the failure remains predominately brittle even at temperatures as high as 1200 °C. The fracture experiments in that study also revealed 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 both parameters decrease with increasing temperature. It was also found that high temperature and low strain rate promote plastic behavior of fine grained Ti_2AlC (Naik Parrikar et al., 2016). Most recently, Bhattacharya and Goulbourne (2016) characterized strain field evolution in coarse grained Ti_2AlC during compressive dynamic loading and showed that macro-scale inhomogeneities in the strain field begin to appear above 200-300 MPa, with some regions even experiencing tension. The latter suggests highly non-uniform stress distribution in the Ti_2AlC polycrystalline microstructure in dynamic loading conditions due to plastic anisotropy of the individual grains.

To facilitate the design and development of Ti_2AlC for various extreme environments, there is a need to understand the dependence of the mechanical response on grain size under different temperatures and loading rates, as the grain sizes would not only affect inhomogeneous stress distribution in the microstructure, but also different deformation mechanisms such as dislocation glide, kinking, delamination and grain boundary sliding. The purpose of this experimental study is to address the aforementioned gap and carry out an experimental investigation of the constitutive behavior of Ti_2AlC under quasi-static and dynamic loading at temperatures ranging from room temperature to 1100 °C, and with different grain sizes.

Material fabrication

Description of sintering process

A two-step process was used to produce high purity Ti_2AlC for the experiments in this study, in which Ti_2AlC powders were first synthesized in-house, then formed into bulk compact material via Spark Plasma Sintering. Ti (99.5%, -325 mesh), Al (99.5%, -325 mesh) and TiC (99.5%, 2 μm) powders for the synthesis of Ti_2AlC powder were purchased from Alpha Aesar, USA. The powder mixture, with a molar ratio of $\text{Ti}:\text{Al}:\text{TiC} = 2:1.05:0.95$, was mixed homogeneously by ball milling for 24 hrs, then placed in alumina boats and sintered in a high vacuum tube furnace (GSL1600X, MTI Corporation, USA) at 1400°C under Ultra High Purity (UHP) argon flow. After drill-milling and sieving the sintered compact of reacted powder, a 170 mesh Ti_2AlC powder was achieved. It was then densified using Spark Plasma Sintering (SPS)¹ (SPS25-10, Thermal Technology LLC, USA) to produce high density samples, by

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¹ Less common but more appropriately referred to as Electric Current Assisted Sintering (ECAS) or Pulse Eclectic Current Sintering (PECS).

sintering for 45 minutes at 1300°C. These specimens are referred to as fine-grained, or FG. To create the larger grain size samples while maintaining an invariant amount of impurities, FG samples described previously were further exposed to heat treatment through a high vacuum tube furnace for periods of 8 to 24 hours, in UHP argon flow. These samples are designated as medium-grained, MG, and coarse-grained, CG, respectively. Through XRD and SEM analysis, it was found that there was only 3-5% TiAl_x impurity by volume, without any TiC and Ti_3AlC_2 ancillary phases formed.

Micrographs of Grain sizes

Fig 1. SEM images of samples with different grain sizes (a) FG, (b)MG and (c)CG

A back-scattered electron detector was used to obtain the microstructure of the prepared specimens during Scanning Electron Microscopy (SEM). Typical SEM micrographs of the polished and etched surfaces are shown in Fig 1, illustrating the different grain sizes and morphologies in as-processed samples. The length and thickness of more than 100 grains for each sample were measured using ImageJ software. Those results showed that on average, grains in FG samples are 6.1 μm long and 4.6 μm thick, in MG samples are 13.9 μm long and 7.3 μm thick and in CG samples are 17.4 μm long and 8.2 μm thick. In addition, the aspect ratio in those samples increased monotonically with increasing grain size, as is expected in MAX phases due to their faster growth in the direction of basal planes.

Experimental setup

Quasi-static characterization

Room and high temperature quasi-static uniaxial compression tests were performed using a servo-hydraulic testing machine (MTS-810, MTS, USA) equipped with a vertical split furnace and SiC pushrods using the procedure detailed in [27]. Cylindrical specimens (5 mm in diameter by 8 mm in length) for the experiments were prepared by wire electron discharge machining (Wire-EDM). Experiments were carried out with fixed crosshead displacement rate to get an initial strain rate of 10^{-4} s^{-1} . A high temperature axial extensometer (632.59E-77, MTS, USA) was attached to SiC spacers to record displacements, and a K-type thermocouple was placed in direct contact with the spacers to monitor the temperature. Heating rates were limited to less than $600 \text{ }^{\circ}\text{C hr}^{-1}$ and a soaking time of 15 minutes was used to ensure that the specimen stabilized at the testing temperature.

Dynamic characterization

Dynamic compression tests at room and high temperature were performed using a modified Split Hopkinson Pressure Bar (SHPB) apparatus (Fig 2). The striker bar, incident bar and transmission bar in the SHPB setup were all made out of Maraging steel. The incident and transmission bars had a diameter of 12.5 mm and lengths of 2133 mm and 1524 mm, respectively. The Ti_2AlC specimens had a diameter of 5 mm and a thickness of 8 mm. The striker bar was propelled using an air-operated gun. Copper pulse shapers of different thicknesses and diameters were used to control the loading pulse. The pulse shaper was placed at the impact end of the incident bar to generate a linear rising compressive pulse. Two tungsten-carbide (WC) inserts were placed between the two bars and the specimen was sandwiched between the inserts. The impedance of the inserts was matched to that of the bars, so that the inserts did not disturb the stress wave profiles. These inserts were used to reduce stress concentration in the specimens and to prevent indentation of the specimens into the bars.

Molybdenum disulfide was used to lubricate the specimen-insert interface to minimize the effects of friction. Single pulse loading of the specimen was ensured by using a setup, proposed by Song and Chen [31], that employs a flange attached to the impact end of the incident bar and an associated rigid mass as a momentum trap. A controlled preset gap is maintained between the flange and the rigid mass, that allows the incident loading pulse to travel but arrests further motion of the incident bar towards the specimen that is caused by the reflected pulse in the incident bar. A Photron SA1 high-speed digital camera was used at a frame rate of 300,000 fps to capture the deformation process. The camera was triggered using an oscilloscope recording strain gage data, resulting in synchronized strain and image measurements.

Fig. 2: Schematic representation of SHPB setup

For high temperature experiments, an induction coil heater was used in conjunction with the SHPB setup. The Ti_2AlC specimens are electrically conductive, and were heated by electromagnetic induction from high current passing through coiled loops around the specimen. The WC inserts prevented the development of a sharp temperature gradient in the bars and also protected the strain gages mounted on the bars. The welding of thermocouples to the specimens was observed to produce minor cracks in them. To avoid this, a calibration procedure was developed where in a specimen with a thermocouple was used to evaluate the amount of power to be supplied to the induction heating system to achieve the desired steady

state temperature in 30 seconds. This calibrated power was used to heat the specimen during experiments, and thermocouple was not attached to specimen directly.

Using one-dimensional wave theory, the strain and stress in the specimen can be determined from the reflected and transmitted strain pulses, respectively, as

$$S_s = E_b \frac{A_b}{A_s} \varepsilon_t(t) \quad (1)$$

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

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, 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:

$$\sigma_s = S_s(1 - e_s) \quad (3)$$

$$\varepsilon_s = -\ln(1 - e_s) \quad (4)$$

Results and discussion

Quasi-static Results

Typical stress-strain curves obtained from quasi-static compression testing at different temperatures are shown in Fig 3. The mechanical response of Ti₂AlC with three different grain sizes is qualitatively similar to the previously reported studies for Ti₂AlC and other MAX phases[32–34]. Below BPTT (~ 900-1000 °C), samples of all grain sizes fail in essentially brittle manner. A very small drop in stress was observed after it reaches maximum value

before the catastrophic failure of the specimens. The fine grained samples exhibit higher compressive strengths as compared to other grain sizes. At 900 °C, the fine and coarse grained samples showed more graceful failure with a larger softening tail after maximum stress to the point of failure. Above BPTT, at 1000 °C and 1100 °C, all samples deformed pseudo-plastically with large strains to failure exceeding $\approx 24\%$ and the difference in compressive strengths between samples with different grain sizes getting smaller. Note here that all samples at 1000 and 1100 °C did not fail at strains of 24%, but the tests were stopped as the upper limit of the extensometer was reached at that point. Above the BPTT, the specimens showed some initial softening after reaching maximum stress, that is, stress decreased with increasing strain, and then transition into small apparent hardening with stresses increasing slightly with increasing strain. The transition point from the softening regime to the hardening regime during deformation above BPTT occurs at higher strains with increasing temperature and decreasing grain size. However, this apparent hardening is most likely an artefact of sample barreling (i.e. significant increase in the cross section area during testing at higher strains) that was not accounted for during calculation of true stresses from the measured loads.

Fig 3. Quasi-static Stress-Strain curves at different temperatures for (a) FG, (b) MG and (c) CG Ti_2AlC .

Dynamic Results

The Ti_2AlC specimens displayed catastrophic brittle failure at room temperature and a brittle graceful failure at high temperatures in dynamic loading conditions. Fig 4(a) illustrates the typical time shifted segments of the recorded pulses while Fig 4(b) shows the corresponding force ratio variation observed for the specimens which exhibited a catastrophic brittle failure. As can be seen in Fig 4(a), the specimen undergoes strain rate acceleration for the first 15 μs causing rising reflected pulse. After 15 μs , and up to the time of failure, the specimen experiences an average strain rate of 400 s^{-1} . The specimen ultimately fails at 38 μs . The occurrence of failure is marked by the peak value of the transmitted pulse, after which the magnitude of transmitted pulse drops sharply. After failure, the magnitude of the reflected pulse rises to the magnitude of the incident pulse. The sharp drop in the transmitted pulse and the rise in the reflected pulse are both characteristic of catastrophic failure. Fig 4(b) shows that the dynamic stress equilibrium (a force ratio of 1) was attained at about 15 μs and was maintained up to the point of specimen failure. The SHPB pulses were analyzed only up to the point of failure (corresponding to peak transmitted pulse) to generate the stress strain curves for the specimens showing catastrophic failure.

Typical pulses and force ratio for a specimen which exhibited a graceful failure are shown Fig 4(c) and Fig 4(d). In this case, the transmitted pulse magnitude decreases gradually upon reaching its maximum in comparison to the catastrophic failure case. The reflected pulse displays a gradual increase in magnitude signifying increase in strain rate as the specimen is strained beyond the peak stress state. The strain rate maintained its constant value up to the point of peak stress. The force ratio plot, Fig 4(d), shows that the specimen is in dynamic equilibrium from 15 μs to 75 μs . The stress strain curves were generated by analyzing the pulses from the start up to the point where the specimen was in stress equilibrium.

Fig 4. Recorded pulses illustrating the difference between catastrophic and graceful failure and force ratio corresponding to these pulses

The true compressive stress-strain curves of fine, medium, and coarse grained Ti_2AlC during dynamic loading at various temperatures are presented in Fig. 5. The dynamic experiments were conducted at an average nominal strain rate of 400 s^{-1} . A '★' symbol is used in these plots to represent points of catastrophic failure. As can be seen in Fig. 5, the peak compressive stress decreases with increase in temperature, similar to what was observed in quasi-static loading, however, that decrease seems to be less significant above BPTT than in quasi-static loading conditions. The specimens had catastrophic brittle failure at room temperature and a more graceful brittle failure at high temperatures. The temperature at which graceful failure

occurs in these specimens decreases with the increase in the grain size. For the dynamic experiments conducted, the graceful failure begins to occur at 1100 °C for fine grain, at 900 °C for medium grain and at 600 °C for coarse grain specimens, which is discussed in more detail in the following section.

Fig 5. Dynamic Stress-Strain curves at different temperatures (a) FG, (b) MG and (c) CG

Compressive strength

The variation of quasi-static and dynamic compressive strength with temperature and grain size is shown in Fig 6. Under quasi-static conditions, the compressive strength is a strong function of grain size below BPTT, in both dynamic and quasi static loading conditions. The specimens having smaller grain size had higher compressive strengths, like that in the case of Ti_3SiC_2 (Radovic et al., 2002) . As testing temperature increases above BPTT, compressive strength of fine grained specimens decreases more rapidly and the difference between the compressive strengths of the samples with different grain sizes diminishes, as seen in Fig 6. This displays good agreement with previous results for MAX phases, namely Ti_3SiC_2 , showing that grain size plays a minor role in the mechanical behavior of these materials above BPTT (Zhen et al., 2005).

The dynamic compressive strengths are higher than the quasi-static compressive strengths with increasing difference between them with increasing grain sizes below BPTT. Under dynamic conditions, the FG specimens displayed greater strength than the large grained

specimens for all the temperatures investigated. Ti_2AlC displays high compressive strength under dynamic loading even at temperatures as high as 1100 °C. Naik Parrikar et al. (2016) have also observed such high compressive strengths in Ti_2AlC under dynamic conditions at temperatures up to 1200 °C. Unlike the quasi-static case, the decreases in dynamic compressive strength with increasing temperature are fairly linear. This indicates different mechanisms of deformation under quasi-static and dynamic behavior at temperatures above BPTT. This higher strength during dynamic loading is more pronounced at higher temperature for all grain sizes investigated here. In addition, although all grain sizes show almost identical compressive strength at 1100 °C in quasi static conditions, the compressive strengths of the samples with different grain sizes still show significant difference in dynamic conditions at the same temperature. It has been reported that the strain rate sensitivity in MAX phases increases with increasing temperature (Radovic et al., 2002). Results presented here suggest that at and above BPTT, fine grain structure shows larger strain rate dependence than coarse grained structures. The latter can be best observed in Figure 7.

Fig 6. Compressive strength as a function of grain size and temperature (a)Quasi-static loading and (b)Dynamic loading

Fig 7. Hall-Petch relation describing compressive strengths as a function of temperatures

(a)Quasi-static Loading and (b)Dynamic Loading

Figure 7 shows Hall-Petch plot where the average compressive strength at various temperatures for the three grain sizes is plotted as function of $l^{1/2}$, where l is the average grain length. The linear regression fits have also been shown in the figure. The results indicate Hall-Petch type relationship between the strength and the grain length under both dynamic and quasi-static loading conditions. The Hall-Petch type relationship between strength and grain length can be associated with failure caused by dislocation pile up on grain boundaries between soft and hard grains that in turn leads to high stress concentrations and fracture (Benitez et al., 2015). In addition, the kinking of hard grains also shows Hall-Petch type dependence with larger grains having higher propensity to buckling and kinking than fine grain ones. Therefore, below BPTT, strains due to easy slip in the soft grains can be accommodated by buckling, kinking and delamination of the hard grains. Stress concentration due to dislocation pile ups is larger and kinking and delamination is easier in the coarse grain structure, so the strength of the coarse grain samples is lower than that of the fine grain ones in both quasi static and dynamic conditions. It was also seen that compressive strength decreases more rapidly below BPTT in quasi static than in dynamic loading conditions. This suggests

that both piling up of dislocations in soft grains as well as kinking and delamination of hard grains are dynamic processes that depend on loading rate.

For quasi-static loading above BPTT, the slope of the linear fitting line decreases significantly in quasi static conditions. Even though the mode of dynamic failure is different above BPTT (more graceful), the Hall-Patch relation is still observed. This suggests that the mechanics governing peak compressive strength and nature of failure (catastrophic or graceful) are different. The peak compressive strength must be driven by the fracture initiation caused by dislocation pileups while the catastrophic or graceful failure must be controlled by the crack propagation and the damage containment characteristics of the material. Another possible explanation for this observation is activation of local grain boundary sliding above BPTT to accommodate stress concentrations as was proposed in (Barsoum and Radovic, 2011) to explain high temperature behavior of MAX phases. Grain boundary sliding is much easier in the fine grain structure making it more sensitive to the loading rate. Stress concentration due to plastic anisotropy of the individual grains is lower in smaller grains, resulting in higher compressive strengths of FG samples in both quasi static and dynamic loading conditions below BPTT compared to the CG structure. Stress concentration can be accommodate easier by grain boundary sliding above BPTT in the FG structure resulting in the more significant drop of the compressive strength in FG structure than in CG one only in a quasi-static loading conditions.

Failure modes

The post mortem specimens were investigated to study the modes of failure. Typical post mortem images of specimens from quasi-static experiments are shown in Fig 8. The specimens from quasi-static experiments tested below BPTT fractured with one or two shear cracks. The angle made by the fractured surface with the loading axis varied from 25°- 36°. Above BPTT, specimens did not fail when tested to strains of 24%, and exhibited extensive

lateral strains and barreling. Shear failure in MAX phases under compression has been reported in a number of studies (Bao et al., 2004; Zhang and Sun, 2005; Bei et al., 2013). During the dynamic experiments, specimens fractured by shear cracks. Post mortem images of specimens from dynamic experiments are shown in Fig 9. The high strain rate allows multiple cracks to grow and hence results in multiple fracture fragments. The difference between catastrophic failure and graceful failure can be clearly seen in these images. During catastrophic failure, a number of small fragments are formed. During graceful failure under dynamic loading, multiple cracks are formed but the bulk of the specimen stays together. Fig 9(i) shows a case where coarse grained Ti_2AlC after 1100 °C did not undergo fragmentation, even though there are multiple cracks in the specimen.

Fig 8. Post-mortem images of specimens after quasi-static loading at 25, 700 and 1000 °C fine grained (a-c), medium grained (d-f) and coarse grained (g-i)

Fig 9. Post-mortem images of FG (a-c), MG (d-f) and CG (g-i) from dynamic experiments

Micrographs

The microscopic images of fracture surfaces from the dynamic compression experiments are shown in Figure 10. Images (a) and (b) depict the specimen fracture surface after dynamic loadings of fine grained and coarse grained specimens at room temperature, respectively. The fracture at room temperature is predominantly intergranular. Images (c) and (d) show fracture surface after dynamic loadings of fine grained specimens at 1100 °C and coarse grained specimens at 900 °C, respectively. Coarse grained specimens at 900 °C were chosen because the specimens at 1100 °C did not fragment. The fracture surfaces have a very rough morphology. The high temperature fracture surfaces show substantial grain refinement. This is due to a combination of cleavage fracture, delamination, and kinking of grains. The kinking of grains at high temperatures leads to higher energy absorption and enables graceful failure under dynamic loading conditions. Similar observations of kinking being operative under dynamic loading and high temperature, have been reported by Parrikar et al. (Naik Parrikar et al., 2016). Several secondary intergranular cracks (marked as →) can be seen in the CG microstructure. These secondary cracks facilitate the kinking of grains and might be responsible for early transition to graceful failure.

Fig 10. Fracture surfaces of specimens from dynamic experiments :(a) FG at 22 °C, (b) CG at 22 °C, (c) FG at 1100 °C and (d) CG at 900 °C

Conclusions

An experimental investigation was conducted to evaluate the effect of grain size on the constitutive behavior of Ti_2AlC under quasi-static and dynamic loading conditions. The results from this study show:

- Under quasi static loading conditions, all 3 grain sized specimens showed brittle failure below 1000 °C (BPTT). At higher temperatures a BPTT was observed.

- Under dynamic loading conditions brittle failure was observed for all three grain sizes for all temperatures (up to 1100 °C).
- A transition from catastrophic failure to graceful failure is observed at higher temperatures, and the propensity of graceful failure increases with increasing grain size.
- The compressive strengths of different grain size materials were related by a Hall Petch relation based on grain length. These Hall Petch relations are valid for temperatures below BPTT for quasi-static loading and valid for temperatures up to 1100 °C for dynamic loading.
- The compressive strengths are governed by dislocation pileup while the nature of deformation, plastic or brittle failure, is governed by crack propagation and crack arrest mechanisms.
- The compressive strengths measured at dynamic strain rates are higher than the strengths measured at quasi-static rates and this difference is more pronounced at higher temperatures.

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Chapter 3

Constitutive Behavior of Ti_3AlC_2 under Dynamic Thermo-Mechanical Loading

by

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Abstract

An experimental investigation was conducted to evaluate the mechanical response of Ti_3AlC_2 under dynamic loading at different temperatures. A Split Hopkinson Pressure Bar (SHPB) apparatus in conjunction with an induction coil heater was used to perform dynamic compression experiments. A series of experiments were conducted at temperatures from 25 °C to 1200 °C at an average strain rate of 500 s^{-1} . Edge notched bend specimens were used to determine the dynamic fracture initiation toughness from ambient temperature to 900 °C with a modified SHPB setup. High speed photography was incorporated to determine fracture initiation time during the dynamic fracture toughness experiments. The results of this study reveal that Ti_3AlC_2 exhibits brittle failure during dynamic loading, even for temperatures of 1100 °C. The peak compressive stress for failure decreased with increasing temperatures. The fracture toughness was also seen to decrease with increasing temperature. The SEM images indicate that under dynamic loading conditions the deformation behavior and fracture mechanisms do not change with temperature.

Introduction

A new class of nano-layered ternary solids called MAX phases have been attracting a lot of attention because of the unique combination of ceramic and metal like properties they exhibit [1–5]. MAX phases are crystalline solids with a general chemical formula of $\text{M}_{n+1}\text{AX}_n$ (where M is an early transition metal, A is an A group element, and X is carbon and/or nitrogen) where M_{n+1}X_n layers interleaved with A-group element layers. MAX phases have high elastic stiffness, good thermal and electrical conductivity, excellent thermal shock resistance, good chemical resistance, high damage tolerance, high fracture toughness, and ease of machinability with conventional tooling [1,2,4–6]. Ti_3AlC_2 belongs to the MAX phase family, and along with the above properties, it also has good irradiation resistance as well as

good resistance to oxidation owing to continuous adhesive Al_2O_3 layer formed on it at high temperatures [7–12].

MAX phases have numerous basal plane dislocations, and only basal plane dislocations can easily move, multiply, and arrange either in pileups or in walls/arrays [13–15], even at temperatures as low as 77 K [16]. This makes MAX phases plastically anisotropic. In addition, MAX phases can also deform by kinking and kink band formation throughout the temperature ranges tested [17–19]. At high temperature, delamination and formation of cavitation and microcracks have also been used to account for observed large strains [20,21]. Under quasi-static loading MAX phases are brittle near room temperature and undergo brittle-to-plastic transition (BPT) at elevated temperatures, usually between 900 - 1100°C [2,4,22–24]. A recent study on fine grained Ti_2AlC showed that its quasi-static response was similar to other MAX phases, undergoing brittle to plastic transition at 900 °C, while under dynamic loading, the response is brittle even at 1200 °C [25]. Fracture toughness studies on MAX phases show that toughness drops significantly at about the brittle-to-plastic transition temperature (BPTT) [26–28]. This suggests that there is no activation of secondary slip systems that contributes to pseudo-ductile behavior of MAX phases above BPTT.

In this article, we present the mechanical response of Ti_3AlC_2 to dynamic loading. The high strain rate compression experiments were conducted in a modified split Hopkinson pressure bar (SHPB) in conjunction with an induction heater for temperatures from 25 - 1200 °C. The dynamic fracture toughness of Ti_3AlC_2 was investigated at 25 °C, 400 °C and 900 °C. These findings will help provide new design considerations for applications where Ti_3AlC_2 may be subjected to dynamic loading. Also, the study will shed light on the influence of lattice parameter ratio, c/a , on the deformation process under dynamic loading.

Experimental procedure

Dynamic constitutive Behavior

Cylindrical specimens of diameter of 6.35 mm and 9.52 mm length were cut from disks by wire EDM. A modified Split Hopkinson Pressure Bar (SHPB) was employed for conducting the high strain rate experiments (Fig. 1). The SHPB setup consisted of an incident and transmitted bar with strain gages mounted at mid span of the bars, as well as a striker bar, all made from Maraging steel. Modifications to the SHPB setup, using inserts and pulse shapers as suggested in [29], were added to account for the hard and brittle nature of Ti_3AlC_2 . Tungsten carbide (WC) inserts were used to sandwich the specimen between the incident and transmitted bars. These inserts prevent the indentation of specimens into the bar, thus avoiding stress concentrations in the specimen and giving correct strain measurements. The impedance of the inserts was matched to the impedance of the bars, hence the inserts did not disturb the stress wave propagation. Copper pulse shapers were used to generate linear rising pulses to accomplish constant strain rate experiments. A flange was attached to the incident bar and in association with a rigid mass, it act as a momentum trap and ensures only a single loading of specimen as explained in [30]. A Photron SA1 high-speed digital camera was used to capture the deformation process during the room temperature experiments. The images were recorded at 300,000 fps and were synchronized with the strain measurements from the SHPB.

Fig. 1: Schematic representation of SHPB setup

Ti₃AlC₂ is electrically conductive, so for high temperature experiments an induction coil heater was used to heat the specimen held within the SHPB setup. Current carrying copper coil loops were placed around the specimen, to heat it using electromagnetic induction. The WC inserts prevented the formation of a sharp temperature gradient in the bars. Spot welding of thermocouples to the specimens was seen to produce minor cracks in them. A calibration procedure was developed to evaluate the amount of power to be supplied to the induction heating system to achieve the desired steady state temperature in the specimen in about 30 seconds. The temperature of the calibration specimen was monitored using a thermocouple spot welded to the specimen itself. During the high temperature experiments, this calibrated heating procedure was used to heat the specimens to the desired temperatures.

Dynamic fracture initiation toughness

Single edge notched bend specimens were used for the fracture toughness (inset in Fig. 2) experiments. The dynamic fracture initiation toughness was investigated at 25 °C, 400 °C and 900 °C. A modified SHPB apparatus with an induction heating system was used to investigate the dynamic fracture initiation toughness (Fig. 2). The apparatus mainly consists of an incident bar, striker bar and pressure gun. The incident bar was made from 9.52 mm diameter brass bar. The strains generated in the incident bar during the dynamic fracture toughness experiments were small and hence were measured using two semiconductor strain gauges (SS-090-060-1150 PB-S1; resistance, $1125 \pm 75 \text{ X}$; gauge factor 155 ± 10 ; Micron Instruments) that were attached in the middle of the bar. The backed semiconductor strain gauges are 75 times more sensitive than the foil type gauges. During loading, the specimen was sandwiched between the incident bar and the rigid frame. For an elevated temperature experiment, the specimen was heated using the induction heater to the desired temperature (usually about 50 °C higher than the test temperature). A thermocouple was attached to the specimen to monitor the specimen temperature. Once the specimen is heated, the incident bar is brought into

contact with the heated specimen and then the striker bar was propelled towards the incident bar using the gas gun. High speed photography was incorporated to identify the time of crack initiation. A Shimadzu HPV-2 high-speed digital camera was used at a frame rate of 500,000 fps with an image resolution of 312 x 260 pixels. The specimen was coated with a white, high temperature paint (VHT SP101) to enhance the contrast of the crack with respect to the specimen. The camera recording was synchronized with strain gauge data to correlate the time of fracture initiation with force measurements.

The load-time history at the specimen/bar interface was 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 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. $K_{ID} = K_I(t_{initiation})$.

Fig. 2: Schematic representation of modified SHPB setup for dynamic fracture toughness evaluation

Results

Constitutive Behavior under dynamic loading

Typical pulses recorded during a room temperature SHPB experiment are shown in Fig. 3. The strain gage on the incident bar measures the incident and the reflected pulses. The copper pulse shaper gives a linear ramp in the incident pulse as seen in the figure. A drastic fall observed in the transmitted pulse, marked by the cross (+), corresponds to the catastrophic failure of the specimen. Similarly, the point marked by the circle (o) on the reflected pulse also corresponds to the specimen's catastrophic failure. Because the strain gages are located at different distances from the edges of the incident bar and transmission bar, the catastrophic failure points in the reflected and transmitted pulses do not overlap with each other in the figure. Beyond the point of catastrophic failure, the magnitude of reflected pulse rises due to the free forward motion of the incident bar end and is not representative of specimen strain. Fig. 4(a) shows the time shifted segments of the recorded pulses that are used for the analysis.

The specimen experiences an average strain rate of 500 s^{-1} and ultimately fails at $51 \text{ } \mu\text{s}$. The force ratio is the ratio of the forces exerted on the two end faces of the specimen. The force ratio evaluated for this experiment is plotted with respect to time in Fig. 4(b). It can be seen from the figure that dynamic stress equilibrium (a force ratio of 1) was attained at about $10 \text{ } \mu\text{s}$ 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 verified for each experiment. 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, A_s is the cross-sectional area of the specimen, and L_s is the thickness of the specimen.

Fig. 3: Typical pulse profiles captured by the strain gages during SHPB experiments

Fig. 4: (a) Time shifted strain gage pulses and (b) force ratio corresponding to these pulses

Fig. 5: Real time images from high speed camera during SHPB experiment

The images that were captured during the room temperature experiments, using the high speed camera, are displayed in Fig. 5. The time of occurrence of these images is marked on the transmitted pulse in Fig 4 with a box symbol (■). Frame 1 in Figure 5 shows the specimen sandwiched between the inserts at the beginning of the experiment, whereas frame 2 shows the compressed state of the specimen, due to relative motion of the WC inserts, at 48 μs before the onset of peak stress. No cracks can be seen in frame 2. At 51 μs (frame 3 in Fig. 5), a macroscopic shear crack can be observed in the specimen, which occurred during the peak stress state in the specimen. Another shear crack can be observed at 55 μs (frame 4 in Figure 5). After the formation of cracks, there is sliding motion along the cracked surfaces resulting

in a rapid decrease of load bearing capacity (sharp drop in transmitted pulse in Figure 3), and complete fracture of the specimen soon after that.

The true compressive stress-strain curves of Ti_3AlC_2 during dynamic loading at various temperatures are represented in Fig 6. The dynamic experiments were conducted at an average strain rate of 500 s^{-1} . The specimens exhibited catastrophic failure at all of the temperatures tested under dynamic loading. The average peak compressive stress in the high strain rate experiments varies from 1260 MPa at room temperature to 780 MPa at 1200°C , and the peak compressive stress decreases with increasing temperature. At high strain rate the specimens show brittle failure while exhibiting high compressive strength. The peak compressive stresses observed during the dynamic experiments are plotted as a function of temperature in Fig. 7. The compressive strength decreases with increasing temperature. The decrease in strength is more rapid at temperatures above 900°C . Even with these high temperatures above 900°C the failure is brittle and the exhibited strength is high (above 700 MPa).

Fig. 6: True compressive stress-strain curve of Ti_3AlC_2 at various temperatures

Fig. 7. Effect of temperature on compressive strength under dynamic loading condition

Dynamic fracture toughness

A typical load-time plot obtained from the strain gage data of a dynamic fracture experiment is shown in Fig 8. Images from the high speed camera for the corresponding experiment used to monitor crack initiation are shown in Fig 9. The crack was seen to grow in the 36th frame, which corresponds to 55 μs as the fracture initiation time on the load-plot in fig 8, represented by the red vertical line in the plot. The load corresponding to fracture initiation time was used to compute the dynamic fracture initiation toughness using equation 2.

Fig. 8: Typical load-time plot measured during a dynamic fracture toughness experiment

Fig. 9: High speed images of the crack during experiment

The dynamic fracture initiation toughness of Ti_3AlC_2 as a function of temperature is shown in Fig. 10. Three experiments were conducted at each temperature and the error bars in Fig. 10 indicate the range of values obtained. The fracture toughness decreases with an increase in temperature. The average dynamic fracture initiation toughness is $13.4 \pm 1.9 \text{ MPa m}^{1/2}$ at room temperature, and is $10.9 \pm 1.4 \text{ MPa m}^{1/2}$ 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 these dynamic experiments.

Fig. 10: Dynamic fracture toughness as function of temperature

Microscopic Images

Fig. 11: Fracture surfaces from experiments, (a) and (b) are from fracture toughness experiments at 25 °C and 900 °C respectively, (c) and (d) are from SHPB experiments

The micrographs of the fracture surfaces from the dynamic experiments are shown in Fig. 11. Images (a) and (b) are fracture surfaces from fracture toughness experiments at 25 °C and 900 °C. The fracture surfaces in both the cases are very similar. The rough fracture surfaces display both intragranular and transgranular failure. The favorably oriented grains seem to have undergone cleavage failure, resulting in flat faces. The refinement grain topography would have resulted from a combination of kinking and grain pullouts. Since there are no

apparent changes in the microstructures, the same mechanisms of deformation must be operational. The drop in fracture toughness at high temperatures is most likely due to ease of dislocation movements at higher temperature. Images (c) and (d) in Fig. 11 are the fracture surfaces from the dynamic SHPB experiments at 900 °C and 1100 °C. The fracture surface morphologies are similar to that of Image (a) and (b), so the mechanism of deformation and crack propagation in Ti_3AlC_2 should be the same for dynamic loading from 25 °C to 1100 °C.

Summary

An experimental investigation was conducted to evaluate the dynamic constitutive behavior and dynamic fracture initiation toughness of Ti_3AlC_2 under thermo-mechanical loading. Ti_3AlC_2 exhibited brittle failure during the experiments from 25 °C to 1100 °C. The peak compressive stress for failure decreased with increasing temperatures. The fracture toughness was also seen to decrease with increasing temperature. The SEM images indicate that under dynamic loading conditions the deformation behavior and fracture mechanisms do not change with temperature.

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Chapter 4

Constitutive behavior of Hastelloy X over wide range of strain rates using Shear Compression Specimen

by

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Abstract

An experimental investigation has been conducted to study the constitutive behavior of Hastelloy X over a wide range of strain rates. Shear compression specimen geometry was utilized to achieve strain rates from 10^{-3} -20000 s^{-1} . A screw driven testing machine and a split Hopkinson pressure bar(SHPB) apparatus were used to load the specimens. An induction coil heating system was utilized in conjunction with SHPB to evaluate constitutive behavior at elevated temperatures under dynamic loading. Room temperature experiments were carried out at varying strain rates from 10^{-3} -20000 s^{-1} , which exhibit the strain rate sensitivity of Hastelloy X. The rate sensitivity increases after rate exceeds 1000 s^{-1} . The stress strain curves at various temperatures under dynamic loading showcase a yield strength anomaly. The stress peaks at the beginning of dynamic yielding at 850 °C and 1000 °C.

Introduction

The objective of this study is to investigate the constitutive behavior of Hastelloy X over a wide range of strain rate (10^{-3} to 20000 s^{-1}). Hastelloy X alloy is a nickel-chromium-iron-molybdenum alloy that possesses an exceptional combination of oxidation resistance, high-temperature strength and fabricability [1]. Hastelloy X is being used in gas turbine engines for combustion zone, industrial furnaces, nuclear plants and chemical process industry [1]. Hastelloy X is also being considered as a skin material for hypersonic flights.

Several studies have been conducted on Hastelloy X. The elastic modulus and quasi-static stress strain curves of Hastelloy X at various temperatures upto 1100 °C can be found in 1998 Military Handbook 5H [2]. Kondo et al. [3] reported how exposure to high temperature helium gas effects the strength and total elongation of Hastelloy X. Brinkman et al. investigated tensile, creep, fatigue, subcritical crack growth, thermal stability properties and the influence of helium environments with controlled amounts of impurities on these properties [4]. Lai [5] investigated the change in the hardness and impact toughness of

Hastelloy X after aging for 10,000 h at temperatures from 538 °C to 871°C. The author found that aging caused M_6C carbides to precipitate which lead to drop in impact toughness. Zhao et al. [6] conducted a study on phase precipitation in Hastelloy X heat-treated at 750, 850 and 900 °C. They observed four different precipitation phases: M_6C , $M_{23}C_6$, σ , and μ and built a time–temperature-transformation diagram for the formation of various phases. Aghaie-Khafri and Golarzi [7] conducted compression experiments on Hastelloy X in the temperature range of 900–1150 °C with strain rates ranging between 0.001 and 0.5/s. They showed that softening mechanisms, dynamic recovery, and dynamic recrystallization occurred during hot working. Sakthivel et al. [8] investigated the Serrated flow behavior of Hastelloy X over a temperature range of 25–750 °C at strain rates between 3×10^{-5} to $3 \times 10^{-3} \text{ s}^{-1}$. They reported that the migration of molybdenum in the nickel matrix is responsible for the occurrence of Portevin-Le Chatelier (PLC) effect in the alloy. Swaminathan et al. [9] utilized Digital Image Correlation (DIC) to study the PLC effect. They found that serrated plastic flow behavior is a consequence of the dynamic strain aging effect and is characterized by strain concentration bands propagating along the length of the specimen. Abotula et al. [10] investigated the dynamic constitutive behavior of Hastelloy X at room and elevated temperatures under strain rates from 1000 to 4000 s^{-1} . They presented the effects of dynamic loading on plastic flow of Hastelloy X for temperatures ranging from 25 to 1100 °C. A number of studies investigating the blast performance of Hastelloy X have also been reported [11–13]. Anomalous yield strength behavior has also been reported during dynamic loading of Hastelloy X [10, 13].

Recently, a specimen geometry, called shear-compression specimen (SCS), has been recently developed by Rittel et al. [14, 15] for evaluation of large strain constitutive behavior over wide range of strain rates. The SCS consists of a cylinder or parallelepiped with a pair of opposite slots machined on its faces. The specimen is loaded axially (either statically or dynamically) and the gage (slot) section undergoes a predominantly shear deformation, with the stresses and strains being rather uniform. Simple relations were developed and assessed to relate the

equivalent true stress and equivalent true plastic strain to the applied loads and displacements [14–16]. Vural et al. [17] conducted an analytical and experimental study of the plastic deformation field in SCS. They proposed modifying the angle of the SCS slot from 45° to 35.26° to enhance the uniformity of stress and strain fields in gage section of SCS. In this study the SCS is utilized to investigate the constitutive behavior of Hastelloy X over a wide range of strain rate (10^{-3} to 20000 s^{-1}).

Experimental procedures

Specimen geometries

The SCS specimens were machined from a 11.11 mm diameter Hastelloy X rod supplied by Haynes International. The geometry of the SCS used is shown in Fig 1. For shear compression tests, all the specimens used in both quasi-static and dynamic tests had a common diameter of 9.53 mm, slot angle of 35° , gage thickness of 2.54 mm and variable gage width of $w = 2.38$, 1.27 and 0.51 mm. The experiments with shear compression specimens were performed over a range of strain rates from 10^{-3} to $2 \times 10^{-4} \text{ s}^{-1}$. For calibrating the SCS, cylindrical specimens with 9.53 mm diameter and 19.05 mm length were prepared and tested in at 10^{-3} s^{-1} strain rate. The equivalent stress (σ_{eq}) and strain in the gage section for the SCS geometry can be expressed in terms of the measured load (P), displacement (d) and the geometrical parameters [14, 15],

$$\sigma_{eq} = k_1 (1 - k_2 \varepsilon_{eq}^p) \frac{P}{Bt} ; \quad \varepsilon_{eq} = k_3 \frac{d}{w} \quad (1)$$

where k_1, k_2, k_3 are constants dependent on material and geometry (w/t). They are determined by comparing the stress strain curves with the curves from cylindrical specimens.

Experimental Setup

Quasi-static experiments were conducted using a screw-driven testing machine (Instron, model 5585). Experiments were carried out on cylindrical specimens and SCS of every gage width at strain rate of 10^{-3} s^{-1} . The SCS were then loaded in the Instron machine to achieve strain rates up to 3 s^{-1} . A Split Hopkinson Pressure Bar (SHPB) was used in the study to achieve higher strain rates up to 20000 s^{-1} . The striker bar, incident bar and transmission bar in the SHPB setup were all made out of Maraging steel. The incident and transmission bars had a diameter of 12.5 mm and lengths of 2133 mm and 1524 mm, respectively. 1018-cold steel pulse shapers were used to control the loading pulse.

High temperature experiments were conducted at strain rates of 5000 and 20000 s^{-1} . The SHPB apparatus in conjunction with the induction coil heating system was utilized as shown in Fig. 2 to conduct experiments up to 1000°C . The temperature of the specimen was monitored using a 0.127 mm chromel–alumel thermocouple, which was spot welded onto the specimen. During the high temperature experiments, the SCS specimen was positioned using two rings made from ceramic foam (2.54 mm thick) as shown in Fig. 2. The bars were kept apart initially, and the SCS was heated in isolation to the desired temperature (usually about 20 C higher than the test temperature). After the specimen achieved desired temperature, bars were brought manually into contact with the specimen. Once the pressure bars were in contact with the specimen the striker was launched using a gas gun.

Fig.1: Schematic representation of SCS (all dimensions in mm)

Fig.2: Fixture to hold SCS during high temperature SHPB experiments

Results

Calibration Experiments

Instron machine was used to perform displacement controlled experiments on cylindrical specimens at a constant strain rate of 10^{-3} s^{-1} to generate reference curve. Then, compression experiments were conducted on SCS with gage width of $w = 2.38, 1.27$ and 0.51 mm . The values of SCS constants that gave the best match with reference stress-strain curve were evaluated. The values of these constants are given in Table 1. The resulting equivalent stress-equivalent plastic strain curves are plotted in Fig. 3. The curves from SCS match well with stress strain curve from cylindrical specimen up to 0.3 strain. These calibrated SCS were then used to evaluate constitutive behavior at higher strain rates.

Table 1: Values of constants for the different gage widths after calibration of SCS

w	k_1	k_2	k_3
2.38	0.75	-0.2	0.55
1.27	0.94	-0.2	0.6
0.51	0.86	-0.2	0.4

A series of experiments were conducted using the Instron machine and SHPB to achieve strain rates from 10^{-3} to 20000 s^{-1} . The SCS enabled achieving strain rates up to 10 s^{-1} . The higher end of strain rates were achieved by choosing a smaller gage width SCS and higher displacement rates in the Instron machine. Similarly, a strain rate of $20,000 \text{ s}^{-1}$ was attained using 0.51 mm wide gage SCS along with incident pulse with a large magnitude. Fig. 4 shows the strain rate variation with time during some selected dynamic experiments using SHPB. The plateaus in the strain rate curves show that the state of constant strain rates achieved during the experiment. The incident pulses were controlled using steel pulse shapers that enable the attainment of constant strain rates. Similar strain rates were maintained for room and elevated temperature experiments as seen in Fig 4. The pulses for higher strain rates have shorter length in time but achieved the same strains because of the higher loading rate.

Fig.3: Comparison between the stress strain curves from the cylindrical specimen and SCS after calibration of constants for SCS

Fig.4: Strain rates as a function of time during dynamic experiments with SCS

Fig.5: Typical stress-strain curves at 25 °C at various strain rates obtained using SCS

The room temperature stress strain behavior of Hastelloy X at various strain rates is shown in Fig. 5. Hastelloy X shows positive strain rate sensitivity. The hardening modulus appears to decrease above strain rates of 5000 s^{-1} . Such softening at high strain rates has been observed in metals in earlier studies [15, 16], which associated this softening behavior to the heat generated from the plastic deformation. Also, the strain rate sensitivity of the material appears to be highly rate dependent and the strain hardening decreases with increasing strain rate. The

flow stress at $\epsilon_{eq}=0.1$ is plotted as a function of strain rate in Fig. 6. Hastelloy X exhibits considerable rate sensitivity, and the rate sensitivity increases beyond 10^3 s^{-1} strain rate.

Fig.6: Strain rate sensitivity of Hastelloy X

Fig.7: Stress–strain curve of Hastelloy X at different temperatures and strain rate 5000 s^{-1}

The effect of temperature on stress strain behavior of Hastelloy X at 5000 s^{-1} is shown in Fig 7. The material shows monotonic reduction in flow stress till $700 \text{ }^{\circ}\text{C}$. At 850 and $1000 \text{ }^{\circ}\text{C}$,

stress was observed to show a peak at yield point, while the flow stress decreased after that. The yield stress at 850 °C was higher than that at 700 °C. This anomalous behavior was also reported by the Abotula et al. [10]. This behavior might be due to the migration of molybdenum atoms and precipitation of molybdenum carbides at high temperature as observed in earlier studies [6, 8].

Conclusions

A series of experiments were conducted using SCS to investigate the constitutive behavior of Hastelloy X at various strain rates. Materials constitutive behavior at elevated temperatures under dynamic loading was also evaluated. The major conclusions of this study are:

- SCS enables investigating the constitutive behavior of Hastelloy X over the range of 10^{-3} – $20,000 \text{ s}^{-1}$ strain rates.
- Hastelloy X exhibited rate dependency. The sensitivity to strain rate increases for strain rates above 1000 s^{-1} .
- At 5000 s^{-1} , the flow stress of Hastelloy X decreased monotonically with increasing temperature up to about 700 °C. At 850 °C and 1000 °C, peaks in stress strain curve are observed at yield point.

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Chapter 5

Response of Curved 2024-Aluminum Panels Subjected to localized Blast Loading at extreme temperatures

by

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Abstract

An experimental investigation was conducted to understand the effect of curvature on blast response of aluminum panels under extreme temperatures. Three aluminum 2024-T3 panels: flat panel and two curved panels, with radii of curvatures of 304.8 mm and 111.8 mm were used for the study. A shock tube apparatus was utilized to impart controlled shock loading. Propane flame torches were used to heat the specimens and experiments were conducted at 25 °C, 200 °C and 350 °C. High speed photography coupled with 3D Digital Image Correlation (DIC) technique was used to obtain full field deflection and velocity, as well as in-plane strain on the back face of the panels. The different modes of deformation and the mode transitions during the blast loading of the three panels have been identified. The effect of temperature on the deformation modes is investigated. The deflections of panel with 304.8 mm radius of curvature were found to be higher than the other two panels. With increase in temperature, the deflections increased and strain was seen to get localized.

Introduction

The objective of this study was to investigate the blast performance of curved 2024 Aluminum panels under extreme thermal environments. 2024 Aluminum is the standard material to build fuselages and lower wing skins [1]. The aluminum components are both flat as well as curved. During adverse conditions, the aircraft components may get exposed to high temperatures. Therefore, in this study, Aluminum panels (flat and two different curvature panels) were subjected to shock loading at room and high temperatures in order to study their dynamic response.

Seidt and Gilat [2] carried experiments on characterizing the plastic anisotropy in 2024 Aluminum over a wide range of temperatures and strain rates. Many studies have investigated the response of metallic plates and beam structures subjected to blast loading [3–9]. Menkes and Opat [3] worked on clamped aluminum beams subjected to shock loading and identified

three damage modes, namely mode-I (inelastic deformation), mode-II (tearing at the extreme fiber) and mode-III (transverse shear at the support). The investigation into these deformation modes were furthered by Nurick et al. [4,5]. Abotula et al. [8] carried shock loading experiments on Hastelloy X at high temperatures up to 900 °C. Kumar et al. [9] conducted experiments and simulations to study the effect of shock loading on curved aluminum panels at room temperature. However, none of the reported studies have looked at the response of curved panels under combined shock and high temperature loadings.

The present study focuses on inelastic deformation of flat and curved Aluminum when subjected to a blast loading under high temperatures. The shock tube was used for shock loading as it provides planar wave fronts and wave parameters that can be easily controlled as seen in previous shock studies [8,10–12]. The results present the evolution of full field deformation captured using digital image correlation (DIC) and influence of curvature on deformation. Different modes of deformation were noticed at various stages of the deformation process. The effect of temperature on deformation magnitude and modes of deformation was also studied.

Experimental Procedures

Material and Specimen Geometry

2024 T3 Aluminum panels were used due to this material's common use on aerospace vehicles. 203.2 x 203.2 x 2 mm flat panels were rolled to two different curvatures to give three sets of specimens; flat (infinite radius of curvature), 304.8 mm radius of curvature, and 111.76 mm radius of curvature. They are further denoted as Panel A, B, and C. The arc length of each plate is 203.2 mm. These panels geometry is noted in Figure 1.

Figure 1: Specimen geometries

Fixture and Shock Tube

Three different fixtures (Figure 2) were used such that each specimen would sit in 12.5 mm wide and 4 mm deep recess between the base plate and top plate with matching curvature. In order to secure the specimen, it was placed in a recess and 2mm spacers were added to the top and bottom edges (along the flat edges) before the top plate was bolted in place. The fixtures shown were then attached to a rigid support at the end of the shock tube such that the shock tube was tangential to each specimen.

Figure 2: Fixtures for holding the specimens

In order to apply the shock load to the specimen, a shock tube was used due to the fact that highly repeatable planar wave fronts could be easily achieved. The shock tube (Figure 3) is made up of a driver and driven section separated by a diaphragm. The driver section is pressurized using high purity helium until the diaphragm ruptures due to the pressure differential. The resultant pressure wave travels down the driven section and develops into a

shock. As the shock wave nears the exit of the tube, two pressure sensors (PCB CA102B) capture the pressure. Typical pressure profiles captured at room temperature by the sensor closest to the panels are shown in Figure 4. It should be noted that the first jump recorded by the sensors is referred to as the incident pressure and the second jump is referred to as the reflected pressure. Typical incident pressures are 0.55 MPa, which caused reflected pressures of 2.2 MPa. The fixture was placed at the end of shock tube such that the center of the 76.2 mm diameter muzzle is oriented tangential to the center of the specimen.

Figure 3: (a) Shock tube schematic (b) Shock tube facility (c) Specimen held tangentially at the muzzle of shock tube

Figure 4: Pressure profiles recorded by the transducer on the muzzle

High Temperature Arrangement

High temperature experiments were carried out at 200 °C and 350 °C. Four propane heating torches were used in order to heat the specimen to the specific temperature. In order to obtain a given temperature, a calibration procedure was undertaken, where a specimen was out-fitted with nine type-k thermocouples in the orientation shown in Figure 5b. The positions and intensity of the torches were adjusted until the desired temperature field at a steady state was achieved. Another thermocouple was used to measure temperatures at each of the corners and edges of the specimen –fixture boundary. An example of a temperature field achieved for a 350 °C experiment can be seen in Figure 5b and the corresponding temperature history until steady state (approximately 1000 seconds) is shown in Figure 5a. When the uniform temperature field was achieved, the nozzles were locked in place and the calibration specimen was removed. For each high temperature experiment, specimens were heated using the calibrated propane torches until the steady-state temperatures were achieved and then the shock loading was applied.

Figure 5: Temperature calibration

High Speed Photography

To capture the deformation event, two high speed cameras, Photron SA 1.1 were used to provide a stereo view of the back-face of the specimen (Figure 6). Many studies have demonstrated the ability of high speed photography in conjunction with DIC to monitor the dynamic deformations during blast loadings. The cameras were used to record each shock experiment at a framing rate of 20,000 fps. Using a high-contrast, randomized speckle pattern, the stereo images could be analyzed using DIC to obtain in-plane, out-of-plane, full field displacements, velocities, and strains. High temperature flame-proof paint was used to apply the speckle pattern to the specimens. To overcome the decorrelation effects from self-radiation of the specimen at high temperature, blue bandpass filters in conjunction with a high intensity light source were used to obtain images with suitable contrast. This technique has been found to be successful in other high temperature DIC investigations [8, 9].

Figure 6: Schematic of the high-speed photography system

Results

Deformation at Room Temperature

The DIC technique was used to obtain full field displacement, strain and velocity data. The evolution of out of plane deflection (does not include initial curvature) and longitudinal strain in the vertical direction, ϵ_{yy} , during the blast wave loading of the panels at room temperature is shown in Fig. 7(Panel A), Fig. 8(Panel B) and Fig. 9(Panel C). In Fig. 7, it can be seen that at 150 μs after the arrival of shock, the panel is deflecting away from shock tube. The central portion has a large deflection due to centralized, plastic deformation which is much larger than the elastic deformation of the rest of the panel. Strain contours show the localized tensile strain in central region. At 300 μs into the deformation process, large plastic deformations reach the boundaries and the similar area of the panel is strained plastically. The deflection images at 400 μs show that the deformation is now influenced by the boundary conditions and the deformation shape transitions from circular to square. The strain contours also change

from elliptical shape to an “X” shape while deforming like a stretched membrane. The panel continues to deform and peak deflection is realized at 600 μs .

Fig. 7: Deflection and strain evolution in Panel A with time

Fig. 8: Deflection and strain evolution in Panel B with time

The deformation and strain contours of Panel B are shown in Fig. 8. 200 μs after the arrival of the shock, the central portion of the Panel B is deflecting away from shock tube, forming an indentation crater in the panel. The portion of the panel near the top and bottom boundaries has deformed towards the shock tube, forming a mode I like deformation shape. Strain contours show the localized tensile strain in central region and compressive strains localized at the top and bottom boundaries. At 350 μs , the central section continues to deform away from shock tube and the size of the indentation crater grows towards the boundaries. The bending of the panel near the top and bottom boundaries resists the deformation and localized bending strains can be seen near the clamped edges. The images at 500 μs show the stage where the panel has been dished out (away from the shock tube) and the strain contours show the membrane stretching tensile strains. The panel continues to deform like a stretched membrane and peak deflection occurs at 850 μs . Fig. 9 shows the deformation process in Panel C. At 200 μs after the arrival of shock, the central portion of the Panel C is deflecting away from shock tube, forming an indentation crater similar to that in Panel B. The elastic waves caused a mode I like deformation shape with the portion of the panel near the top and bottom boundaries moving towards the shock tube. Strain contours show the bending strains in the panel, tensile strain in central region, and compressive strains getting localized at the top and bottom boundaries. The indentation crater in the central section grows till 950 μs and deforms the panel away from shock tube. The bending of the panel near the top and bottom boundaries resists the deformation and localized bending strains can be seen near the clamped edges. The strain contours show that as the indentation crater grows, large compressive strains get localized at the clamped boundary edges.

Fig. 9: Deflection and strain evolution in Panel C with time

Fig. 10: Evolution of deflection over the vertical line slice at center of the panel: (a) Panel A, (b) Panel B and (c) Panel C

Vertical line slices at the center of the panels were inspected to further understand the deformation process and the evolution of their deflection is shown in Fig. 10. The 0 μ s line represents the undeformed configuration before the arrival of the shock. The deformation in all the panels begins with indentation of the central region away from the shock tube. In Panel A, Fig. 10(a), the indentation mode of deformation changes to a global membrane like stretching at 400 μ s. In Panel B, the indentation deformation leads to a mode I shape with two valleys around the indentation as seen at 150 μ s in Fig. 10(b). As the Panel deforms further, the indented region grows and moves away from the shock tube and the two valleys are pushed towards the boundaries. The panel section from the clamps to the valley act like cantilever arms resisting the motion of the indented region. By 500 μ s the indentation region reaches the clamped boundaries and causes Panel B to dish out, away from the shock tube. Panel B is then stretched like a membrane and peak deformation occurs at 850 μ s. Panel C deforms in an indentation mode. When the central indentation zone in panel C grows, the region in the clamped boundary curves into a smaller curvature and resists the indentation growth like cantilever beams. This leads to localization of strains in the clamped ends as seen in fig 9.

Fig. 11: Time history of (a) deflection and b) velocity at the center point of the three panels

The progression of deflection and velocity at the center point on the three panels is shown in Fig. 11. The deflection-time plot, Fig. 11(a), shows that the deflection of Panel B is the 30mm, the highest among the three panels. Panel A and Panel B, both initially deformed in indentation mode and then transitioned into membrane like stretching of the panel, while panel C has only indentation deformation. The dishing of Panel B following the indentation deformation caused a larger deflection magnitude in comparison to the other panels. The velocity-time plots of Panel A and Panel B show two distinct peaks in velocity. The first peak in the velocity plot corresponds to the peak velocity attained during the indentation mode (at 150 μ s in Panel A, 150 μ s in Panel B and 250 μ s in Panel C) and the second peak in the velocity is during the membrane like stretching of Panel A and Panel B. During the membrane stretching deformation phase of Panel A and Panel B, as the deformation increases, more area of the Panels resists the deformation and hence a sharp deceleration is observed after the peak in the velocity. In Panel C, which deforms in indentation mode, the area resisting the deformation decreases with the growth of indentation, hence the deceleration on the panel to zero velocity is slower.

Deformation at High Temperature

Fig. 12: Deflection history of the center point of the three panels at (a) 200 °C and (b) 350 °C

The evolution of out of plane deflection at the center point of the three panels under high temperature shock loading is shown in Fig. 12. The modes of deformation of the three panels remain the same as those during room temperature. The deformations increase with the increase in temperature. These larger deformations can be explained by the decrease in yield strength of Aluminum with increase in temperature. Panel A and Panel B deflections at high temperature increased by same the magnitude from the room temperature, while the increase in deflection of Panel C is more. In Panel C, as the temperature increases, the size of indentation also increases leading to a larger increase in deflection at higher temperatures. Fig. 13 shows the deformations across vertical line slices on the three panels at various temperatures during peak deflection, along with the undeformed configuration. The full field of longitudinal strain in the vertical direction, ϵ_{yy} , in the three panels at 350 °C is shown in Fig. 14. The propane torches blocked some of the speckle pattern and areas near them could not be analyzed by DIC. The strains at 350 °C were higher than those noticed during room temperature experiments. The tensile stretched regions are seen to get localized towards the corners in Panel A and Panel B at 350 °C. In Panel C, at 350 °C the higher tensile strains are seen near the valley as compared to the central region at room temperature experiments. This is due to the additional stretching of the indented zone at 350 °C. The region near the clamped boundary of Panel C shows large values of compressive strains formed while resisting the growth of indentation zone. The vertical slice across Panel C at 350 °C in Fig. 13(c) shows the bulging of the indented region is prominently higher than at 200 post-mortem images of the shock loaded aluminum panels and room temperature. With increase in temperature, the deflections increase throughout the panels as shown in Fig. 13. The increase in deflections is due to the higher strains produced at higher temperatures.

The deformation of Panel C shows that deformation mode is a strong function of initial curvature. The yield strength of aluminum panels decreased with increasing temperature, but

the deformation modes did not change. The decrease in yield strength resulted in localization of strain and increase in deformation magnitudes.

Fig. 13: Deformed shape of the vertical line slice at center of the panels at various temperatures: (a) Panel A, (b) Panel B and (c) Panel C

Fig. 14: Strains at peak deformation of the panels at 350 °C

Post Mortem Images

The post-mortem images of the shock loaded aluminum panels are shown in Fig. 15. Panels A and B show stretching in a X shaped manner whereas panel C exhibits indentation deformation. The deformations are more pronounced at high temperature (Fig. 15 (d-f)). In Panels A and B creases are observed that separate the tensile and compressive regions of the panels. Panel C had an elliptically shaped indentation at room temperature (Fig. 15(c)) and rectangle like indentation at 350 °C. Also, X shaped creases can be seen in the indented region of Panel C at 350 °C.

Fig. 15: Post-mortem images of panels: (a-c) at 25 °C and (d-f) at 350 °C

Conclusions

Localized blast loading experiments were conducted on three Aluminum panels with varying curvatures at 25 °C, 200 °C and 350 °C. High speed photography coupled with 3D DIC technique was used to study the evolution of full field deflections and velocity and in-plane strain on the back face of the panels. The results from the study show:

- Localized blast loading leads to indentation mode of deformation. As the deformation magnitude increases, the deformation can transition into global dishing of the panel.
- As the radius of curvature decreases, the panels tend to resist the blast loading by deforming in indentation mode only.
- The curvature of the panel is more important governing factor in the deformation mode in comparison to the yield strength or loading magnitude.

- The panels that underwent dishing deform by membrane stretching and the panels that deform by indentation mode only resist deformation by bending of the region near clamped edges.

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Appendix A

Investigation of Dynamic Shear Banding in Ti6Al4V alloy using Digital Image

Correlation

by

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Introduction

Adiabatic shearing is a phenomenon characterized by a localization of the plastic deformation in narrow bands. The mechanisms of these adiabatic shear bands formation was first explained by Zener and Hollomon [1]: in metallic materials, a large proportion of the plastic deformation energy is converted into heat. During a dynamical loading, as the deformation duration is very short, the heat transfers by conduction in the material are negligible. The consequence of this effect is a local growth of the temperature in these bands, a thermal softening of the material and a local increase of the plastic deformation. Generally, intense plastic shear strain in the band is the precursor of voids growth and crack initiation and propagation. All the mechanisms of initiation and propagation are not completely known today [2].

The formation duration of an ASB is about 10 μs and the band width is around 10 μm which poses many challenges in the design of a measurement devices. Marchand and Duffy [3] used a bar of 12 InSb detectors to visualize on the torsion specimen twelve zones of 35 μm by 35 μm and spaced of 11 μm . The maximum temperature reached 590 °C in a HY100 steel. Liao and Duffy [4] carried shear banding experiments on the titanium alloy Ti6Al4V with a space resolution of 17 μm , and measured maximum temperatures between 440 °C and 550 °C. Guduru et al. highlighted the two-dimensional character of the ASB by visualizing the temperature field at a distance of 5.5 mm of a crack point during the shear band formation.

They reported that the temperature distribution along the band is highly non-uniform with discrete regions of high temperature.

Recently, a specimen geometry, called shear-compression specimen (SCS), has been recently developed by Rittel et al. [5, 6] for evaluation of large strain constitutive behavior over wide range of strain rates. The SCS consists of a cylinder or parallelepiped with a pair of opposite slots machined on its faces. The specimen is loaded axially (either statically or dynamically) and the gage (slot) section undergoes a predominantly shear deformation, with the stresses and strains being rather uniform. Simple relations were developed and assessed to relate the equivalent true stress and equivalent true plastic strain to the applied loads and displacements [5–7]. Vural et al. [8] conducted an analytical and experimental study of the plastic deformation field in SCS. They proposed modifying the angle of the SCS slot from 45° to 35.26° to enhance the uniformity of stress and strain fields in gage section of SCS. Rittel and Wang [9] have studied the thermo-mechanical aspects of adiabatic shear band (ASB) formation for two alloys: Mg AM50 and Ti6Al4V. Tests were carried out on shear compression specimens (SCS) using SHPB apparatus. The evolution of the temperature in the deforming gauge section is monitored at a single point, using an array of high-speed infrared detectors. In this study we seek to evaluate the strain distributions during adiabatic shear band formation with higher temporal and spatial resolution. Dynamic loading was conducted on SCS of Ti6Al4V alloy and the strain fields generated were studied by utilizing high speed images and digital image correlation (DIC).

Experimental Procedures

The SCS specimens were machined from a commercially available 9.53 mm diameter Ti6Al4V rod. For shear compression tests, all the specimens used had a common diameter of 9.53 mm and gage width of $w = 1.27$ mm. In order to control the point of origin of shear

bands a slot, 0.25 mm wide and 0.49 mm long, was machined at the end of the gage section using an end mill. The geometry of this slotted SCS is shown in Fig. 1.

Fig.1: Schematic representation of SCS with slot (all dimensions in mm)

A Split Hopkinson Pressure Bar (SHPB) was used to apply dynamic load to Ti6Al4V SCS specimens. The striker bar, incident bar and transmission bar in the SHPB setup were all made out of Maraging steel. The incident and transmission bars had a diameter of 12.5 mm and lengths of 2133 mm and 1524 mm, respectively. The striker bar was propelled using an air-operated gun. Molybdenum disulfide was used to lubricate the specimen insert interface to minimize the effects of friction. A high-speed digital camera (Hyper Vision HPV2, Shimatzu) was used at a frame rate of 500,000 fps and an exposure time of 1 μ s to capture the deformation process. High temperature flame-proof paint was used to apply the speckle pattern to the gage section of the specimens. Formation of shear bands is accompanied by high temperatures. To overcome the decorrelation effects from self-radiation of the shear bands at high temperature, blue bandpass filters in conjunction with a high intensity light source were used to obtain images with suitable contrast. This technique has been found to be successful in other high temperature DIC investigations [10, 11]. The camera image capturing was

triggered from the oscilloscope recording strain gage data, resulting in synchronized strain and image measurements. The high speed images were analyzed using a 2 dimensional DIC software, Vic 2D, to obtain full field displacement and strain data.

Results and discussion

Fig.2: Typical strain pulses recorded during dynamic experiments

Typical strain pulses recorded by the strain gages mounted on the pressure bars, during the dynamic experiments at 6000 s^{-1} strain rate are shown in Fig. 2. The duration of transmitted pulse is shorter than that of the incident and reflected pulse. The sharp drop in the transmitted pulse represents the failure of specimen due to formation of shear bands. When the specimen fails, the incident pulse undergoes reflection from a free face. Hence the magnitude of reflected pulse reaches the magnitude of incident pulse after the failure of specimen.

The full field strain images before the point of complete fracture and the corresponding transmitted pulse during a dynamic experiment is shown in Fig. 3. The von-mises strain contours are uniform till $76 \mu\text{s}$. After that strain gets localized at the top right corner of the gage. SCS geometry causes small stress concentrations at gage boundaries and hence are more

susceptible to formation of shear bands. The shear band is formed at top right corner and progresses along the gage boundary from right to left. Localized high strains are seen in the vicinity of shear band, which finally leads to fracture of the specimen.

Fig.3: (a) Transmitted pulse during dynamic experiment and (b) In plane Von Mises strain contours in the gage section

Fig.4: Strain contour in the gage section on SCS with a slot at the end

A slot was introduced at the end of the gage section to initiate the shear banding in front of the slot. This helped in better visualization of the shear banding event. Fig. 4 shows von-mises strain contours observed during experiment with a slotted SCS. The formation of non-uniform strain bands can be seen at 52 μ s of deformation. The high strain values are significantly away from the end of the slot, suggesting this localization is due the material deformation characteristics. The slot just allows the control of the region where localization occurs, thus aiding in better visualization of the strain field formed. As the deformation proceeds, the localization of strains increases. A fracture initiates from the end of the slot, but much higher strain values can be seen ahead of the slot. The strain values of three points, P1, P2 and P3,

marked in fig. 4 are plotted in Figure 5 along with the transmitted pulse. The plateau in the transmitted pulse indicates the beginning of plastic deformation of the gage. The strain values at the three points are the same till 46 μs . After that they begin to vary differently. The strain values rise rapidly, which is an indication of formation of shear band. These strain values are plotted until the point where crack growth caused decorrelation.

Fig.5: Transmitted pulse and strains at select locations on the gage for a slotted SCS

Summary

Dynamic loading of Ti6Al4V was conducted in SHPB apparatus. SCS were used to enable visualization of the shear banding phenomenon. The gage section of SCS was speckled and high speed photography was used to capture the deformation event. The DIC analysis of the high speed images shows localizations in strains. The introduction of slot in gage section helps to control the region where shear banding occurs.

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