Effect of milling time on the compressive high strain rate behavior of Al-SiC Composite

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Abstract. Aluminum-Silicon carbide (Al-SiC) metal matrix composite (MMC) have gained importance as a new class of material capable of serving the future generation brake material requirements. The presented study deals with the compressive high strain rate behavior of Al-SiC composite comprising 15% SiC by weight. The MMC specimens were fabricated by powder metallurgy route. Three different ball milling times were selected for the study purpose after optimizing all other processing parameters. Incorporation of SiC invariably enhanced the hardness, quasi-static and dynamic compressive strength of Al-SiC composite. Hardness was noted to enhance as a function of increasing ball milling time. However, the quasi-static and dynamic compression properties were recorded highest for the ball milling time of 240 minutes. The study confirms that minor addition of a suitable reinforcing material can significantly improve the properties of an MMC. Also, increasing the ball mill time may not necessarily enhance all the material properties.

1. Introduction
Composites have emerged as a new class of material systems that can be tailored to suit the specific property requirements. In general, three different types of composites are available namely, metal matrix composites (MMC), ceramic matrix composites (CMC) and fibre reinforced composites (FRC). FRCs are primarily used for the applications demanding low density and high strength; CMCs are limited to high-temperature applications; MMCs are extensively used for the property enhancement of metals by addition of suitable reinforcement. Incorporation of high strength, high hardness or high melting point materials in a metal matrix enhances various properties of the resulting composite. Aluminum is one such metal which in general possesses low strength and low hardness. Incorporation of suitable hard material in the Aluminum matrix may result in significantly higher hardness and strength of the resulting Aluminum metal matrix based composite.

Numerous studies have been reported in the literature citing the uses of different reinforcing materials for the property enhancement of Aluminum matrix composite. Patidar et al. [1] in their review works reported significant improvement in the properties of Aluminum based MMC as a function of increasing boron carbide. Previtali et al. [2] worked with SiCp and B4Cp as reinforcement with A359 Aluminum as the matrix material and reported that incorporation of 20% SiCp results in better properties compared to 7.5% B4C addition. Behm et al. [3] reported improvement in the high
strain rate performance of Al-B4C composite. Shen et al. [4] worked on Al-7075/B4C composites fabricated by Powder metallurgy route and reported that incorporation of B4C beyond 7.5% will not enhance the MMC performance. Zhang et al. [5] reported in their works on Al-SiC composites fabricated by powder metallurgy technique that lower reinforcement size enhances the compressive high strain properties of composite and the strain softening in the dynamic stress–strain curves is credited to the adiabatic heating. Rammath et al. [6] in their review works confirmed that incorporation of SiC is better over Al2O3 and B4C for higher wear resistance. Mocko et al. [7] worked with A359/SiCp composite and reported enhancement in the compressive high strain rate properties. In the light of available literature, it can be concluded that incorporation of SiC is better over other ceramics as far hardness and wear resistance are concerned. Aluminum-Ceramic based composites are primary candidate materials for brake drums. Since at times brakes may be loaded under high rates of loading, therefore, there is a strong need to study the high strain rate behavior of Aluminum Silicon carbide based MMC. The strain rate of loading refers to ‘rate of change of strain’. Rate dependency of majority of materials is reported in the known literature [8]. Zhang et al. [9] reported that the rate-dependent behavior of Al under high strain rate loading in the range of 600 /s to 7000 /s results in enhanced material properties.

Presented work deals with the compressive high strain rate behavior of Aluminum-Silicon carbide (Al-SiC) MMC made by powder metallurgy process. The SiC powder weight percent was kept constant at 15% and the ball milling time was varied to study the effect of milling time on MMC properties. The green powder compacts were compressed on a hydraulic press and sintering was done in argon inert atmosphere. The Al-SiC composite specimens thus produced were tested for micro hardness, quasi-static compressive strength and dynamic compressive high strength. For compressive high strain rate loading split Hopkinson pressure bar (SHPB) apparatus was used. Three different milling times were studied for comparison and one set of pure Aluminum was used as the reference material.

2. Material and Samples
A commercial grade of Aluminum powder (Make: Lobe Chemie, Product code: 00880) having 98% purity at 325 mesh size and Silicon Carbide powder (Make: Chemical Drug House, Product code: 024354) with 95% powder passing through 400 mesh were used for the sample preparation. The melting and boiling point of Aluminum powder used are 660.37 °C and 760 °C, respectively. For specimen preparation, 85% Al and 15% SiC powder by weight were ball milled. The powder to ball weight ratio for ball milling was set to 1:5. For better mixing of powders, two different sizes of steel balls were used. In the present case, 4 balls of 18 mm and 60 balls of 6.35 mm diameter were used for the mixing of powders. 2wt% stearic acid was added to act as a process control agent. The rotational speed was optimized to 100 rpm prior to the preparation of final samples. Rotation time of 120, 240 and 360 minutes was used for the preparation of three different type of Al-SiC samples. A die was fabricated for the preparation of solid cylindrical specimens having a diameter of 12.8 mm and the thickness of the specimen was controlled by varying the weight of powder. For green compacts preparation, metal powder was compacted in a hydraulic press at 100 kN for 240 seconds. Three different Al-SiC green powder compacted sample sets were prepared for studying the rate dependent material behavior. Also, the Al powder as provided was milled for 240 minutes to prepare samples that can serve as a reference for studying the enhancement achieved due to the addition of SiC.

Knowing the melting temperature of Al powder, the sintering temperature was set just above 90% the melting point. To protect the specimens from oxidation at high temperature, argon gas was used. The flow rate of argon was set to 6 lit/min from the initiation of the sintering process till the end. All the specimens were sintered for 180 minutes at 600 °C, followed by cooling inside the muffle furnace.

The composite samples dimensions thus prepared were in accordance with the recommendations of literature (Gama et al. 2014). To maintain the aspect of 0.5 for high strain rate testing of a specimen having a diameter of 13 ± 0.2mm, the specimen thickness was set to 6.5 ± 0.2mm, respectively.
3. Experimental

Universal testing machine (UTM) and split Hopkinson pressure bar (SHPB) apparatus were used for measuring the quasi-static and dynamic compressive behavior of Al-SiC composites. The UTM used for the compression test was Instron make having 100kN load cell and the in-house developed SHPB was based on 16 mm diameter Titanium bars. The rate of compression on UTM was set to 5 mm/min in accordance with the ASTM-E9 standard and in case of SHPB, the rate of loading is controlled by nitrogen gas pressure. For micro hardness determination, Vickers hardness tester was used at 10kg load.

The in-house developed SHPB apparatus comprises of nitrogen-based gas gun for imparting the velocity to the striker bar as shown in figure 1. The striker bar, in turn, hits the incident bar which results in the generation of compressive stress wave. The stress wave on reaching the other end of incident bar interacts with the specimen. A part of stress wave travels through the specimen into the transmission bar and partly the stress wave is reflected back. The reflected part of the stress wave is responsible for the assessment of specimen strain and the stress wave recorded by the strain gauge mounted on the transmission bar gives the specimen stress. The other end of transmission bar hits the hydraulic oil filled damping system which serves as a momentum trap. A suitable signal conditioner and amplifier and data acquisition system are used for the collection of raw data using Labview software, which is later processed using Matlab to reveal the stress-strain behavior using Eq. (1) - (3).

Incorporation of suitable pulse shaper is recommended in the literature [11]. Linatex, a natural rubber having 1.3 mm thickness was used as a pulse shaper. The complete specifications of developed SHPB are available in the literature [12][13].

Figure 1. Schematic arrangement of a typical compressive SHPB apparatus.

1-dimensional wave propagation in the elastic bars serves as the basis for the determination of stress, strain and strain rate induced in the specimen. The SHPB was designed and fabricated in accordance with the scheme originally suggested in the literature by Kolsky (1949). The necessary mathematical formulations for the composite properties determination are established in the known literature and are given as (Gama et al. 2014),

\[ \dot{\varepsilon}_s(t) = \left( \frac{2C_0}{L_S} \right) \varepsilon_r(t) \]  
(1)

\[ \varepsilon_s(t) = \pm \left( \frac{2C_0}{L_S} \right) \int_0^t \varepsilon_r(t) \, dt \]  
(2)

\[ \sigma(t) = \pm \frac{E_A}{A_S} \varepsilon_r(t) \]  
(3)

4. Results and Discussion

Al powder comprising 15% SiC were milled for three different times for the fabrication of Al-SiC composites after optimizing all other relevant parameters. For the comparison of the results, one set of a sample of pure Al was milled for 240 minutes. After sintering of composite specimens, micro
hardness testing was done on the Vickers hardness testing unit. Quasi-static testing of cylindrical specimens was done on UTM and compressive high strain rate testing was done on compressive SHPB.

4.1 Quasi-static testing
Quasi-static compression of all the material compositions under consideration was done at a constant rate of loading of 5 mm/min. Gradual loading of pure Al and Al-SiC composites revealed a difference in the compressive behavior as a function of milling time. Figure 2 depicts the test results of quasi-static compression of all the material samples till the strain of 0.5. Pure Al-based specimen was expected to perform not as good as SiC reinforced and the same was confirmed by quasi-static testing. The unexpected result is the lower stress of 360 min. milled Al-SiC composite compared to other lesser milled specimens. On the basis of quasi-static test results, it is obvious that 240 min. milling time is optimum for the material and processing scheme under consideration as revealed from figure 2.

The initial void compression is recorded highest for the specimen milled for highest time, depicted in the form of strain growth with insignificant stress enhancement for 360 min. milled Al-SiC. Relative lesser initial non-linear growth of stress indicates optimum powder size achieved after milling for 240 min. From figure 2 it is evident that 240 minutes of milling time for Al powder comprising 15% SiC is the best.

![Figure 2. Compressive quasi-static behavior of developed Al based composites](image)

4.2 Micro hardness testing
The micro hardness testing was done on Vickers micro hardness tester using 10kg load. Table 1 shows expected results in the form of higher micro hardness as a function of increasing milling time. In the case of pure Aluminum, milling resulted in minor improvement in the micro hardness. Incorporation of 15% SiC significantly enhanced the micro hardness of sintered Al-SiC specimens. The average micro hardness of five samples is presented in Table 1 and a consistently increasing micro hardness trend is recorded as a function of increasing ball milling time. The increase in hardness due to increasing milling time is attributed to the finer powder size resulting in better packaging of powder specimens. However, it may be noted that overall change due to variation in the milling time is insignificant. Therefore, on the basis of micro hardness test results, no inference should be drawn for the finalization of milling time.

4.3 High strain rate compressive testing
The cylindrical composite specimens were kept in between the incident and transmission bar and due to striker bar impact on the incident bar, a compressive stress wave travels through both the bars and specimen. The signal conditioner, amplifier and data acquisition system ensures recording of a change in voltage as a function of strain variation, which in turn is used to estimate specimen strain, stress and strain rate using Eq. (1) – (3). The set-up is calibrated in accordance with the scheme suggested in the literature Naik et al. (2008) before conducting the experiments.
Table 1. Effect of ball mill time on hardness.

| S. No. | Material | Mill time (min.) | Hardness (HV10) |
|--------|----------|------------------|-----------------|
| 1      | Aluminum | Nil              | 31.4            |
| 2      | Aluminum | 240              | 34.3            |
| 3      | Al-SiC   | 120              | 46.8            |
| 4      | Al-SiC   | 240              | 47.8            |
| 5      | Al-SiC   | 360              | 48.2            |

Figure 3 depicts the rate dependent stress-strain behavior of pure Aluminum and Aluminum comprising 15% SiC under identical loading conditions. Non-reinforced specimens resulted in minimal stress within the range of strain rate of loading. An increasing strain rate of loading from 1600/s to 2800/s enhanced the peak stress by 24%. Incorporation of 15wt% SiC significantly enhanced the compressive high strain rate performance of Al-based composite.

Figure 3. Stress-strain plots of compressive high strain rate loading of (a) 240 min. milled Al; (b) 120 min. milled Al-SiC; (b) 240 min. milled Al-SiC; and (b) 360 min. milled Al-SiC.

All the stress-strain curves irrespective of the rate of loading and reinforcement addition revealed an initial linear stress growth followed by strain hardening in the plastic zone. Increasing rates of loading enhanced both the initial linear section and strain hardening controlled plastic deformation zone. All the specimens revealed hardening as a function of strain, which in literature is reported as strain-induced hardening and the same is held responsible for stress growth after an initial linear stress growth [16]. When the loading cycle is close to decay, the specimen stress falls suddenly, indicating negligible damage towards the end of the loading cycle. It may be noted that all the specimens were recovered intact without any macroscopic failure within the experimental range of loading. Increasing milling time enhanced peak stress till 240 min. of mill time. Further higher mill time diminished the high strain rate performance of Al-SiC composite.
Under identical loading conditions, different strain rates were attained by the specimens derived from the same material composition. This can be attributed to two main factors responsible for strain softening: (i) adiabatic heating resulting in significant temperature increase under high rate of loading and (ii) microstructure damage due to dynamic compression. This adiabatic heating not only varies strain but also controls the stress growth after the initial linear stress growth. The stress range for 120 min. milled Al-SiC composite was in the range of 194 – 261 MPa within the strain rate of loading ranging from 1300 /s - 2600 /s, respectively. Similarly, the peak stress attained by 240 min. and 360 min. milled Al-SiC composite was in the range of 203 – 264 MPa and 214 – 250 MPa for the strain rate of loading ranging between 1515 /s – 2485 /s and 1360 /s – 2650 /s, respectively.

It may be noted that 240 min. milling time resulted in the peak performance of the composite under consideration. This may be attributed to the optimal ball mill time, as higher mill time results in finer powder but associated with mill time is a possibility of oxidation and cold welding. Though process control agent is added to limit cold welding yet the same may occur at an insignificant level. Oxidation is always a possibility as the ball milling process was not accomplished under a fully controlled environment. Lower ball milling times on the other hand will not permit powder to reach the optimum size.

Figure 4 shows the effect of increasing strain rate of loading on the strain at peak stress, peak stress and toughness. The strain growth as a function of the growing strain rate of loading was depicted quite identical for all the materials under consideration (Figure 4 (a)). Only 240 min. milled Al-SiC underwent slightly strain at peak stress at higher strain rates of loading. All other compositions resulted in perfectly linear growth of strain as a function of increasing strain rates of loading. Linear strain growth at peak stress is approximated with help of linear regression and the resulting mathematical equations are presented in Eq. (4) – (7).

\[
\begin{align*}
\text{Al 240 min.} & \quad \varepsilon_s = 5E-05\dot{\varepsilon}_s + 0.0374 \quad (R^2 = 0.9734) \\
\text{Al-SiC 120 min.} & \quad \varepsilon_s = 6E-05\dot{\varepsilon}_s + 0.0052 \quad (R^2 = 0.9573) \\
\text{Al-SiC 240 min.} & \quad \varepsilon_s = 7E-05\dot{\varepsilon}_s - 0.0147 \quad (R^2 = 0.9993) \\
\text{Al-SiC 360 min} & \quad \varepsilon_s = 6E-05\dot{\varepsilon}_s + 0.0175 \quad (R^2 = 0.9991)
\end{align*}
\]

Similar to strain growth, peak stress was also noted to enhance as function of growing strain rates of loading, as shown in figure 4 (b). Importance of ball milling in terms of stress improvement as function of rate of loading clearly indicates better performance of 240 min. milled composites compared to any other material under consideration. The linear regression capable of predicting the stress behaviour as a function of strain rate of loading is given in Eq. (8) – (11).

\[
\begin{align*}
\text{Al 240 min.} & \quad \sigma = 0.03\dot{\varepsilon}_s + 124.95 \quad (R^2 = 0.9503) \\
\text{Al-SiC 120 min.} & \quad \sigma = 0.0498\dot{\varepsilon}_s + 126.6 \quad (R^2 = 0.9737) \\
\text{Al-SiC 240 min.} & \quad \sigma = 0.0579\dot{\varepsilon}_s + 120.09 \quad (R^2 = 0.9493) \\
\text{Al-SiC 360 min.} & \quad \sigma = 0.0302\dot{\varepsilon}_s + 170.72 \quad (R^2 = 0.9546)
\end{align*}
\]

Toughness (Y) plays a vital role in case of impact loading and the same was noted maximum for 120 min. milled Al-SiC composite. However, toughness being governed by the area under the stress-strain curve is higher due to the higher total strain of 120 min. milled composite. Also, the insignificant difference in the slope of toughness rise may be noted for 120 min. and 240 min. milled specimens from figure 4(c) and Eq. (13) and (14), respectively. Toughness growth can be estimated using linear regression for all the material systems using Eq. (12) – (15). It may be noted that the coefficient of regression for all the cases under consideration is quite satisfactory. Therefore, prediction of material properties can be done using the linear regression equations.
Al 240 min.  
\[ Y = 0.0114\dot{\varepsilon}_s - 0.3997 \ (R^2 = 0.9718) \]  
(12)

Al-SiC 120 min.  
\[ Y = 0.0214\dot{\varepsilon}_s - 15.134 \ (R^2 = 0.9800) \]  
(13)

Al-SiC 240 min.  
\[ Y = 0.0209\dot{\varepsilon}_s - 14.268 \ (R^2 = 0.9847) \]  
(14)

Al-SiC 360 min.  
\[ Y = 0.0169\dot{\varepsilon}_s - 6.9744 \ (R^2 = 0.9903) \]  
(15)

5. Conclusions

The presented experimental study deals with the high strain rate compressive behaviour of Aluminum and Aluminum composite comprising 15wt% of Silicon carbide. Incorporation of SiC invariably enhanced the high strain rate performance of Al based composite. Following conclusions are drawn on the basis of experimental study:

i) Increasing rate of loading enhanced the compressive properties of both Al and Al-SiC composite. Compared to pure Al, incorporation of SiC enhanced rate dependent compressive strength by 28%.

ii) Ball milling time can vary the performance of both pure metal as well as metal powder based composites. The material performance enhanced as the mill time enhanced from 120 min. to 240 min. However, further higher mill times could not add to the performance of Al-SiC composite under high strain rate loading.

iii) Maximum stress associated with minimum total strain for 240 min. milled Al-SiC powder based specimens confirm the need for optimization of mill time.

iv) The hardness improvement as a function of 15wt% SiC incorporation was 39%. Therefore, incorporating trace of suitable hard elements can further enhance the hardness.

Figure 4. Effect of strain rate on (a) strain at peak stress; (b) stress; and (c) toughness.
Acknowledgments
Authors are grateful to the Mechanical Engineering Department, IIT Delhi for granting the permission to conduct the experiments.

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