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Synthesis And Characterization Of Novel Ni-Max And Alsi-Max Composites

Maharshi Dey

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SYNTHESIS AND CHARACTERIZATION OF NOVEL NI-MAX AND ALSI-MAX COMPOSITES

by

Maharshi Dey

Bachelor of Technology (ME), West Bengal University of Technology, 2014

A Thesis
Submitted to the Graduate Facility
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Grand Forks, North Dakota
December
2018
This thesis, submitted by Maharshi Dey in partial fulfillment of the requirements for the Degree of Master of Science from the University of North Dakota, has been read by the Faculty Advisory Committee under whom the work has been done and is hereby approved.

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This thesis meets the standards for appearance, conforms to the style and format requirements of the School of Graduate Studies at the University of North Dakota, and is hereby approved.

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Department Mechanical Engineering
Degree Master of Science

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ABSTRACT

The main objectives of this thesis are: (a) synthesize and characterize Ni-Ti$_3$SiC$_2$ composites (Chapter 2), (b) understand and compare the effect of other MAX phases like Cr$_2$AlC, Ti$_3$AlC$_2$ and MoAlB on the mechanical and tribological behavior of Ni-matrix composites (Chapter 3), and (c) explore an alternative alloy to compare the tribological performance with Ni-Matrix composites (Chapter 4). This study shows that Ni-matrix composites can be fabricated by using a combination of cold pressing and subsequent hot pressing. All the composites were then studied by detailed microstructure investigations, mechanical, and tribological characterizations.
CHAPTER I

INTRODUCTION

1.1 SOLID LUBRICANTS AND NICKLE MATRIX COMPOSITES

Solid lubricants have become important engineering materials for reducing wear and friction coefficient between rotating components. Its potential usage can eliminate or reduce the reliance of oil-based machinery needed for oil lubrication. CaF$_2$, h-BN, WS$_2$, and graphite are some examples of solid lubricants which are currently being studied as additives in metal alloys for solid lubrication [1].

Ni is an important metal for designing different types of structural alloys. The demand for Ni has increased over the years. Amongst all, China has also become the largest consumer and importer of Nickel [2]. Similarly, Ni-matrix composites have also emerged as an important source for designing structural component due to their high mechanical strength couple with high wear resistance [3]. Hard particles like TiC can improve the wear resistance of pure Ni [3]. Ni-Matrix composites are also attractive as solid lubricants with additives like Ag, CaF$_2$/BaF$_2$ eutectic for high performance applications like foil bearings [4, 5]. A critical aim of this thesis is to explore novel solid lubricants, more particularly MAX phases as solid lubricants, in Ni-matrix composites (please see next section for details).

1.2 MAX PHASES

MAX Phases are layered, hexagonal, early transition-metal ternary carbides and nitrides [6]. Their chemical formula is $M_{n+1}AX_n$, where $n = 1, 2$, or $3$, M is an early transition metal, A is a group A element, and X is carbon or nitrogen (Fig. 1.1) [6-8]. These phases have characteristics reminiscent of both metals and ceramics, for example, they are excellent thermal and electrical temperatures. However, like a ceramic, it is oxidation resistant, extremely refractory, and able to
maintain strength at elevated temperatures [6]. Different groups have reported promising behavior of properties of MAX-metal composites [8-11]. Kothalkar et al. [8] reported an enhancement by 1.5 times in yield strength in AA6061/Ti$_2$AlC composites as compared to peak aged AA6061. Kothalkar et al. [9] also studied equiatomic NiTi and Ti$_3$SiC$_2$ and observed extraordinarily high damping in both thermo-mechanically cycled and as-sintered composites until 200 MPa. Agne et al. [10] presented a fundamental study that Al-V$_2$AlC composites can be fabricating by quenching rather than furnace cooling. The research group at University of North Dakota led by Prof. Gupta has explored the use of these phases as particulates in metal and polymer matrices [12-19]. conductivity, easily machinable, relatively soft, resistant to thermal shock, and plastic behavior at higher

![Figure 2.1: MAX phases are made up of M – early transition metal (red), A – element from the A groups (blue), and X – carbon or nitrogen (black). Their chemical formula is M$_{n+1}$AX$_n$, where n = 1, 2, or 3, and each respective group is known as 211, 312, or 413 [7]](image-url)
1.3 MAX REINFORCED METALS (MRMS)

Metal matrix composites that are reinforced with MAX phase will be referred to MAX reinforced metals (MRMs) in this thesis. In previous studies, the addition of MAX phases has shown improvement in the mechanical and tribological properties of different metal matrices like Al, Sn, Zn, Ag, and Bi [12-15]. The beneficial effects of adding Ti₃SiC₂ to various metal matrices is summarized in the Figs. 1.2 and 1.3 [15]. MAX phase particulates have also shown beneficial effects on the mechanical and tribological performances in polymer matrices [19-21]. These composites are called MAX reinforced polymers (MRPs). These can be used in potential applications where excellent tribological performances of polymer matrix composites are desired. The main objectives of this thesis are: (a) synthesize and characterize Ni-Ti₃SiC₂ composites (Chapter 2), (b) understand and compare the effect of other MAX phases like Cr₂AlC, Ti₃AlC₂ and MoAlB on the mechanical and tribological behavior of Ni-matrix composites (Chapter 3), and (c) explore an alternative alloy to compare the tribological performance with Ni-Matrix composites (Chapter 4).

Figure 1.2: Plot of stress versus displacement of yield strength of different MRMs versus Ti₃SiC₂ content [15].

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Figure 1.3: Plot of MRM s (a) $\mu_m$, and (b) wear rates as a function of Ti$_3$SiC$_2$ content [15].
CHAPTER II

SYNTHESIS AND CHARACTERIZATION OF NOVEL NI-Ti\textsubscript{3}SiC\textsubscript{2} COMPOSITES

This work has been published in the Proceeding of the 42\textsuperscript{nd} International Conference on Advanced Ceramics and Composites, Ceramic Engineering and Science Proceedings Volume 39, Issue 2, 2018, “Synthesis and Characterization of Novel Ni-Ti\textsubscript{3}SiC\textsubscript{2} Composites”, M. Dey, M. Fuka, F. AlAnazi, and S. Gupta.

2.1 INTRODUCTION

Nickel is an important material for various engineering applications due to its unique properties like magnetism, strength, corrosion resistance, and wear resistance [1]. The wear resistance of Ni can be further improved by adding dispersed hard particles like SiC, TaC, and NbC into the Ni matrix [1-3]. These composite materials and coatings are promising materials for protection of tribological components, low cost coatings for replacement of hard Chromium coatings, among other applications [1-3]. Ni-matrix composites have also attracted a lot of attention for their potential usage as solid lubricants by incorporating various additives like Ag, and CaF\textsubscript{2}/BaF\textsubscript{2} eutectic for high performance applications like foil bearings [4-6]. Based on these results, it can be hypothesized that lubricious particulates like MAX Phases can be promising additive for enhancing the functional attributes of the Ni and Ni-based alloys.

As a background, it is now well established that M\textsubscript{n+1}AX\textsubscript{n} (MAX) phases, where n =1,2,3; M is an Early Transitional Metal, A is a Group A element (mostly groups 13 and 14); and X is C and/or N, are novel ternary carbides and nitrides which have excellent properties like damage
tolerance, thermal shock resistance and machinability [7-11]. Gupta et al. [12-15] have showed that the addition of MAX phase (Ti_3SiC_2) particulates have enhanced the mechanical and tribological performance of Al-, Sn-, Zn-, Bi- and Ag-matrix composites. The authors have classified these new generation of composites as MRM (Metal Reinforced with MAX) as 5-30 vol% Ti_3SiC_2 was used to reinforce the metal matrix. A major hurdle for designing Ni-Ti_3SiC_2 composites is the propensity of reaction between Ni and Ti_3SiC_2 phases at 1450 °C during the sintering process [16]. The goal of this paper is to report the synthesis and characterization of novel Ni-Ti_3SiC_2 composites at lower temperatures by hot pressing.

2.2 EXPERIMENTAL

Ti_3SiC_2 powder (-325 mesh, Kanthal, Hallstahammar, Sweden) and calculated concentrations of Ni powders (Part number 43214 (-325 mesh and 99.8% pure), Alfa Aesar, Haverhill, MA) were dry ball milled (8000 M mixer Mill, SPEX SamplePrep, Metuchen, NJ) for 2 minutes. All the mixed powders were then poured in a die and then hot pressed (HP) under a uniaxial compressive stress of ~240 MPa (~12.7 mm die) at 600 °C for 5 minutes. For comparison, samples of pure Ni were also fabricated by following the above-mentioned procedure at ~240 MPa (Ni (240 MPa)) and ~165 MPa (Ni (165 MPa)), respectively. Ni-based MRM composites were designed by adding 5 vol% (Ni-5%312Si), 10 vol% (Ni-10%312Si), 20 vol% (Ni-20%312Si) Ti_3SiC_2, and 30 vol% (Ni-30%312Si) Ti_3SiC_2 in the Ni- matrix. Theoretical density of Ti_3SiC_2 and Ni particulates were used to calculate the theoretical density (ρ_T) of all the composite samples by using the rule of mixture calculations. The experimental density (ρ_E) of the composites was then calculated from the mass and dimensions of each sample. Thereafter, the porosity (P) of the sample was determined by the Eq. 1.

\[ P = \left(1 - \frac{\rho_E}{\rho_T}\right) \times 100 \]
For each composition, a set of 5 samples were then tested in compression by using a mechanical testing unit (Shimadzu AD-IS UTM, Shimadzu Scientific Instruments Inc., Columbia, MD) at a deflection rate of 1 mm/min on cube samples (~3 mm x ~3 mm x ~3 mm) which were machined by a diamond saw from the hot-pressed samples.

Due to experimental limitations, stress versus displacement plots are reported as the actual strain during mechanical testing could not be determined. In the paper, the yield strength is defined when stress versus displacement plot transitions from the linear to non-linear regime. The linear region of the composites had a regression fitting of $R^2 > 0.95$. For each composite, an average of 5 yield strength measurements is reported in the text [12-15]. Vicker’s micro-hardness indentor (Mitutoyo HM-112, Mitutoyo Corporation, Aurora, IL) was performed by loading the samples at 4.9 N for 12 s. In the text, an average of at least five readings for each composite system is reported. XRD measurements were performed on the samples by using a Rigaku Diffractometer (SmartLab, Rigaku, Japan) at a scan rate of 4 °/min from $20^\circ$ to $50^\circ$. The tribological behavior of the samples were investigated by using a ball-on-disc tribometer (CSM Instruments SA, Peseux, Switzerland) against alumina ($\text{Al}_2\text{O}_3$ balls (~6 mm diameter), Anton Paar USA Inc., Ashland, VA) and stainless steel (SS) (100Cr6 (~6 mm diameter), Anton Paar USA Inc., Ashland, VA). The experiment conditions used during these studies were 5 N, ~31.4 cm/s linear speed, ~5 mm track radius, and a sliding distance of 200 m and 1000 m for testing the composites against SS and $\text{Al}_2\text{O}_3$ balls, respectively. All the Ni-$\text{Ti}_3\text{SiC}_2$ disks were also polished to a ~1 µm finishing which were confirmed by using a surface profilometer (Surfcom 480A, Tokyo Seimitsu Co. Ltd., Japan). After performing the tribological testing for each composition, the $\mu_{\text{mean}}$ was then calculated by averaging the mean results obtained from the three and five data sets of similar Ni-$\text{Ti}_3\text{SiC}_2$ composition during sliding against SS and $\text{Al}_2\text{O}_3$, respectively. The mass of the samples and
substrates were measured before and after the testing by using a weighing scale (Model XA82/220/2X, Radwag Balances and Scales, Poland). The specific wear rate (WR) was calculated from:

\[
WR = \frac{(m_i - m_f)}{(\rho . N . d)} \tag{II}
\]

where, \(m_i\) is the initial mass, \(m_f\) is the final mass, \(\rho\) is density of the composite, \(N\) is the applied load, and \(d\) is the total distance traversed by the sample during the tribology testing. The total WR from both the counterparts is reported in the text. The methodology for inspecting the tribosurfaces are reported in Chapters 2 and 3.

2.3. RESULTS AND DISCUSSION

2.3.1 MICROSTRUCTURE AND PHASE ANALYSIS

SEM images of Ni-Ti₃SiC₂ composites are shown in Fig. 1. In all the compositions, Ti₃SiC₂ particulates are well-dispersed in the Ni matrix. The inspection of the interface between Ni and Ti₃SiC₂ particles even at higher magnifications did not show any discernible interfacial reaction (Fig. 1f). XRD plots of Ni-Ti₃SiC₂ composites also show predominantly Ni and Ti₃SiC₂ peaks (Fig. 2). By analyzing the Figs. 1 and 2, it can be construed that Ti₃SiC₂ particulates have minimal reaction with the Ni-matrix. Further TEM studies are recommended to study the interfaces of Ni and Ti₃SiC₂ particulates (currently, this facility is beyond the experimental capability of the authors). Figure 3 plots the variation of porosity as a function of Ti₃SiC₂ content. When the Ti₃SiC₂ content was varied between 5 and 20 vol%, the Ni-Ti₃SiC₂ composites had similar porosity of ~12%, for example, Ni-5%312Si had a porosity of ~11.8% and Ni-20%312Si had porosity of ~12.5%. Comparatively, Ni and Ni-30%312Si fabricated under similar conditions had porosity of ~9.82% and ~17.7%, respectively. For comparison, Ni (165 MPa) had a porosity of ~17.7% (Fig.
3a). In general, samples fabricated by hot pressing showed higher density than the samples fabricated by pressureless sintering [12-15]. The hardness of Ni-Ti$_3$SiC$_2$ composites retained similar values of ~1.58 GPa in Ni to ~1.53 GPa in Ni-5%312Si although the former sample was denser, thereafter it decreased from ~1.47 GPa in Ni-10%312Si to 1.26 GPa in Ni-30%312Si (Fig. 3b). Among all the MRMs studied so far, Ni-based MRMs showed the highest hardness (Fig. 3c).

2.3.2 COMPARISON OF MECHANICAL PERFORMANCE OF MRMS

Figures 4 shows the plot the compressive strength versus displacement plots of different Ni-Ti$_3$SiC$_2$ composites. At lower concentration until ~10 vol% of the Ti$_3$SiC$_2$ additions, the addition of Ti$_3$SiC$_2$ particulates in the matrix compressive did not have any appreciable effect on the mechanical performance, but after addition of higher concentration of Ti$_3$SiC$_2$, the failure become more brittle in nature. Figure 4b summarizes the yield strength of Ni-Ti$_3$SiC$_2$ composites for better understanding of the mechanical behavior of these composites. More particularly, the mean yield strength of Ni-Ti$_3$SiC$_2$ composites varied from ~600 MPa in Ni to ~592 MPa in Ni-5%312Si, thereafter, it decreased gradually to ~465 MPa in Ni-20%312Si, and then it sharply decreased to ~227 MPa in Ni-30%312Si. Comparatively, the yield strength of Ni (165 MPa) was ~467 MPa. Clearly, porosity and Ti$_3$SiC$_2$ are both contributing to the mechanical performance, and more studies are needed to delineate the effect of these two factors. Comparably, the yield strength of the other MRMs was significantly lower (Fig. 4c). These results show that by tailoring the particulate content and the metal matrix, it is quite possible to engineer the mechanical behavior of these composites according to the strength requirement for a specific design.
2.3.3 SUMMARY OF TRIBOLOGICAL BEHAVIOR OF DIFFERENT MRMS

Figures 5a and 5b show the plot of friction coefficient ($\mu$) versus distance of Ni-Ti$_3$SiC$_2$ composites sliding against alumina and SS balls, respectively. While sliding against Al$_2$O$_3$, Ni-Ti$_3$SiC$_2$ composites attained steady state after cycling for ~200 m although Ni-30%312Si showed some fluctuations (Fig. 5a). Comparatively, Ni-Ti$_3$SiC$_2$ composites sliding against SS balls showed greater fluctuations, but by qualitatively inspecting the plots, it can be surmised that the addition of Ti$_3$SiC$_2$ has increased the $\mu$ (Fig. 5b). For detailed analysis, Figs. 6a and b plot the WR and $\mu$ as a function of Ti$_3$SiC$_2$ content. During sliding against SS, the addition of Ti$_3$SiC$_2$ particulates marginally decreased the WR from ~0.003 mm$^3$/N.m in Ni to ~0.002 mm$^3$/N.m in Ni-10%312Si, thereafter the composites retained similar WR values until Ni-30%312Si (Fig. 6a). The $\mu_{\text{mean}}$ was ~0.55 in Ni, thereafter, it retained similar value of ~0.54 in Ni-5%312Si, and then it increased to ~0.78 in Ni-10%312Si. Thereafter, the $\mu_{\text{mean}}$ increased to ~0.86 in Ni-30%312Si (Fig. 6b). In other words, this study shows that the addition of ~5 vol% Ti$_3$SiC$_2$ can marginally reduce the adhesive wear in Ni-Ti$_3$SiC$_2$/steel tribocouple without affecting the $\mu$. More studies are needed to optimize the particle size and concentration of Ti$_3$SiC$_2$ particulates as higher amount of Ti$_3$SiC$_2$ additions (>10 vol%) increased the $\mu$ without having drastic effect on the WR.
Figure 2.1: SEM SE micrograph of, (a) Ni, and BSE images of, (b) Ni-5%312Si, (c) Ni-10%312Si, (d) Ni-20%312Si, (e) Ni-30%312Si, and (f) higher magnification of the region marked in (e).

Figure 2.2: XRD profiles of, (a) Ni-5%312Si, (b) Ni-10%312Si, and (c) Ni-20%312Si
Figure 2.3: Plot of, (a) porosity, and (b) hardness versus Ti$_3$SiC$_2$ content, and hardness versus Ti$_3$SiC$_2$ content of different MRMs studied in a previous work [15].

Figure 2.4: Plot of, (a) stress versus displacement profile of Ni-Ti$_3$SiC$_2$ composites, and (b) yield strength versus Ti$_3$SiC$_2$ content.
Comparatively, during sliding against Al$_2$O$_3$, Ni had a WR of $\sim 2.95 \times 10^{-5}$ mm$^3$/N.m, and the WR marginally decreased to $\sim 2.42 \times 10^{-5}$ mm$^3$/N.m in Ni-5% Si, thereafter the WR increased gradually to $\sim 3.96 \times 10^{-5}$ mm$^3$/N.m in Ni-10% Si, and then increased sharply to $\sim 1.78 \times 10^{-3}$ mm$^3$/N.m and $8 \times 10^{-4}$ mm$^3$/N.m in Ni-20% Si and Ni-30% Si, respectively (Fig. 6a). The $\mu_{\text{mean}}$ was $\sim 0.36$ in Ni, thereafter, it gradually increased to $\sim 0.54$ in Ni-10% Si, and retained similar values until Ni-30% Si (Fig. 6b). This study shows that the addition of $\sim 5$ vol% Ti$_3$SiC$_2$ can marginally reduce the abrasive wear without drastically affecting the $\mu$. The addition of 10 vol% and higher concentration of Ti$_3$SiC$_2$ increased both the WR and $\mu$. It was shown in the last section that the additions of Ti$_3$SiC$_2$ makes the composites brittle as compared to the pristine Ni matrix (Fig. 4b). The higher WR at $>10$ vol% Ti$_3$SiC$_2$ addition can be explained by the inherent brittleness of the composite which renders it more vulnerable to the abrasive wear.

![Figure 2.5](image): Plot of friction coefficient (\(\mu\)) versus distance of Ni-Ti$_3$SiC$_2$ tribocouples against, (a) alumina, and (b) SS.
2.3.4 STUDY OF TRIBOFILMS AND MECHANISM OF WEAR

Figure 7 shows the SE SEM micrographs of the Ni-10%312Si (Figs. 7a-b) and alumina surface (Figs. 7c-d) after tribology testing. Both the surfaces are covered with tribofilms which are created due to the wear and subsequent oxidation of Ni-10%312Si surface, for example, the presence of sub-stoichiometric micro constituents *Ni$_{0.87}$Al$_{0.06}$Ti$_{0.06}$Si$_{0.02}$O$_{0.61}$C$_{x}$* (Fig. 7b) and *Ni$_{0.92}$Al$_{0.03}$Ti$_{0.03}$Si$_{0.01}$O$_{0.25}$C$_{x}$* (Fig. 7d) on Ni-10%312Si and alumina surface, respectively. By analyzing the tribochemistry of SEM images, it can be construed that the Ni-based MRMbs are mainly contributing to the formation of tribofilms. Figure 8 shows the tribosurfaces of SS (Figs. 8 a-b) and Ni-5%312Si (Figs. 8 c-d). Both the surfaces showed Ni-rich sub-stoichiometric oxides which shows that mainly Ni-Ti$_3$SiC$_2$ composite surface is contributing to the formation of tribofilms. According to the classification developed by Gupta and Barsoum [17] tribofilms can be classified as Type IV tribofilms where MAX based composites contribute to the formation of tribofilms. Based on the architecture of the tribofilms, Gupta and Barsoum [17] further divided the Type IV tribofilms into several sub-categories, namely: (a) Type IVa tribofilms (lubricous and
chemically homogenous at the microscale) [17], (b) Type IVb tribofilms (decomposed Ag-rich regions mixed with different triboxides) (Fig. 12b) [17], and (c) Type IVc tribofilms (powdery) [12]. The tribofilms formed in Ni-Ti₃SiC₂ and alumina or stainless steel tribocouples are lubricious (when the Ti₃SiC₂ content is ≤10 vol%), thus by using the classification above, the tribofilms can be classified as Type IVa.

![SEM micrographs](image)

Figure 2.7: SEM micrographs of, (a) Ni-10%312Si (SE), (b) BSE image of the same region, (c) alumina surface, and (d) alumina surface at higher magnification (BSE).

In summary, the addition of 5-10 vol% of Ti₃SiC₂ particulates has some beneficial effect on the tribological behavior without having an adverse effect on the mechanical behavior thereafter both the tribological and mechanical properties detoriate at higher concentration of Ti₃SiC₂ particulates. Table 1 summarizes the tribological behavior of different Ni-based solid lubricants. The WR of well-studied solid lubricant compositions like PS300, PS400, and NAF 5 lies in the range of 10⁻³-10⁻⁵ mm³/N.m. These solid lubricants are also composed of multiple constituents (Table 1). Clearly, Ni-Ti₃SiC₂ composites offer a new alternative for Ni-based solid lubricants.
with the inherent advantage that the composition is simpler, and can be potentially deposited into coatings. More studies are needed to understand the high temperature lubrication behavior of these composites.

Figure 2.8: SEM micrograph of, (a) SS surface (SE), (b) BSE image of the same region, (c) Ni-5%312Si surface (SE), and (d) BSE image of the same region.
Table 1.1: Summary of tribological behavior of different Ni-based coatings and composites

<table>
<thead>
<tr>
<th>Coatings /Compositions</th>
<th>Countersurface</th>
<th>Conditions</th>
<th>WR</th>
<th>µ</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>PS300 (NiCr-Cr₂O₃ (80.3 vol%), Ag (5.5 vol%), CaF₂/BaF₂ (14.2 vol%))</td>
<td>Al₂O₃ (pin)</td>
<td>Pin-on-disk, RT, 4.91 N, and 1 m/s</td>
<td>~2.3 x 10⁻⁴ (total wear)</td>
<td>0.62</td>
<td>4</td>
</tr>
<tr>
<td>PS400 (NiMoAl (70 wt%)-Cr₂O₃ (20 wt%)-Ag (5 wt%)-BaF₂/CaF₂(5 wt%))</td>
<td>Inconel X-750 (pin)</td>
<td>Pin-on-disk, RT, 4.91 N, and 3 m/s after previous sliding at 500 °C and 650 °C</td>
<td>~1.1 x 10⁻³ (total wear)</td>
<td>0.31</td>
<td>5</td>
</tr>
<tr>
<td>NAF5 (Ni-alloys–12.5%Ag–5%BaF₂/CaF₂)</td>
<td>Si₃N₄ ball</td>
<td>Ball-on-Disk, RT, 5 N, and 1 m/s</td>
<td>~6 x 10⁻⁵ (WR of NAF5)</td>
<td>0.3</td>
<td>6</td>
</tr>
<tr>
<td>NAF10 (Ni-alloy-12.5%Ag-10%BaF₂/CaF₂)</td>
<td>Inconel 718</td>
<td>Ball-on-Disk, RT, 5 N, and 1 m/s</td>
<td>~7 x 10⁻⁵ (WR of NAF10)</td>
<td>0.32</td>
<td>6</td>
</tr>
<tr>
<td>NAF15 (Ni-alloy-12.5%Ag-15%BaF₂/CaF₂)</td>
<td>Inconel 718</td>
<td>Ball-on-Disk, RT, 5 N, and 1 m/s</td>
<td>~10⁻⁴ (WR of NAF15)</td>
<td>0.35</td>
<td>6</td>
</tr>
<tr>
<td>NAF5 (Ni-alloys–12.5%Ag–5%BaF₂/CaF₂)</td>
<td>Inconel 718</td>
<td>Ball-on-Disk, RT, 5 N, and 1 m/s</td>
<td>~5 x 10⁻⁵ (WR of NAF5)</td>
<td>0.32</td>
<td>6</td>
</tr>
<tr>
<td>NAF10 (Ni-alloy-12.5%Ag-10%BaF₂/CaF₂)</td>
<td>Inconel 718</td>
<td>Ball-on-Disk, RT, 5 N, and 1 m/s</td>
<td>10⁻⁴ (WR of NAF10)</td>
<td>0.34</td>
<td>6</td>
</tr>
<tr>
<td>NAF15 (Ni-alloy-12.5%Ag-15%BaF₂/CaF₂)</td>
<td>Inconel 718</td>
<td>Ball-on-Disk, RT, 5 N, and 1 m/s</td>
<td>~8 x 10⁻⁵ (WR of NAF10)</td>
<td>0.35</td>
<td>6</td>
</tr>
</tbody>
</table>
2.4. CONCLUSIONS

Ni-Ti$_3$SiC$_2$ composites were fabricated by hot pressing. The microstructure evaluation showed that Ti$_3$SiC$_2$ particulates are well dispersed in the microstructure with minimal interfacial reactions. The mean yield strength of Ni-Ti$_3$SiC$_2$ composites varied from ~600 MPa in Ni to ~592 MPa in Ni-5\%312Si, thereafter, it decreased gradually to ~465 MPa in Ni-20\%312Si, and then it sharply decreased to ~227 MPa in Ni-30\%312Si. Ti$_3$SiC$_2$ particulates had a beneficial effect on the tribological behavior. More particularly, it was observed that the addition of 5-10 vol\% Ti$_3$SiC$_2$ can help in mildly reducing both the adhesive and abrasive wear, but at higher concentrations, the composites were found to be more vulnerable towards abrasive wear as compared to adhesive wear due to brittle nature of the composites. The tribofilms formed during the dry sliding of Ni-Ti$_3$SiC$_2$ composites against both SS and alumina were further investigated by SEM and X-ray analysis and were further classified into Type IVa tribofilms.

2.5. Acknowledgements

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CHAPTER III

SYNTHESIS AND CHARACTERIZATION OF NOVEL Ni-MAX COMPOSITES

3.1 EXPERIMENTAL

Ti$_3$AlC$_2$ powders were fabricated by mixing powders of TiH$_2$ (Part number 209279 (-325 mesh and 98% pure), Sigma-Aldrich, St. Louis, MO), Al (Part number 11067 (7-15 micron powder and 99.5% pure), Alfa Aesar, Haverhill, MA) and TiC (Part number 40178 (~2 micron powder and 99.5% pure), Alfa Aesar, Haverhill, MA) in 1:1:2 molar ratio. The powders were then dry mixed for 5 minutes in a ball mill (8000 M mixer Mill, SPEX SamplePrep, Metuchen, NJ) in a methacrylate vial with methacrylate balls. The powders were further mixed with 10 wt% PVA for 10s. The mixed powders were then poured in a ~25.4 mm die and then cold pressed (Model 3853-0, Carver, INC., Wabash, IN) at ~88 MPa to make compact pellets. The pellets were then annealed at 750 °C for 2h and then heated up to 1400°C for 2h at a heating rate of 10 °C/min. Similarly, Cr$_2$AlC powders were fabricated by mixing Cr (Part number 266299 (-325 mesh and ≥99% trace metal basis), Sigma-Aldrich, St. Louis, MO), Alumina (Lot KO5Z034 (-325 mesh, 99.5% trace metal basis), Alfa Aesar, Haverhill MA), and Carbon (LotY19B065 (Graphite powder crystalline, (-325 mesh and 99% pure)), Alfa Aesar, Haverhill MA) in a molar ratio of 2:1:1:1. The powders were then ball milled for 5 mins in a methacrylate vial with methacrylate balls. The mixed powders were then poured in a ~25.4 mm die and cold pressed (Cold press, model 3853-0, Carver, INC., Wabash, IN) at ~88 MPa to make compact pellets. This pellet was then heated at 1350 °C for 4 h.
The sintered pellets of both compositions were then ball milled (8000 M mixer Mill, SPEX SamplePrep, Metuchen, NJ) for 30 min in an 8001B hardened steel vial with 8001B hardened steel balls. The resultant powders were then sieved until -325 mesh in a sieve shaker. XRD measurements, performed by a Rigaku Diffractometer (SmartLab, Rigaku, Japan) at a scan rate of 4 °/min from 20° to 50° confirmed single phase Ti$_3$AlC$_2$ and Cr$_2$AlC powders.

In this study, 5 vol%, 10 vol%, 20 vol%, and 30 vol% of Ti$_3$AlC$_2$ and Cr$_2$AlC particulates were used as a reinforcement in the Ni-matrix composites. They will be referred to by using the following nomenclature, Ni-xvol%Ti$_3$AlC$_2$ or Ni-xvol%Cr$_2$AlC. For example, Ni-20vol%Cr$_2$AlC means that Ni matrix is reinforced with 20 vol% Cr$_2$AlC. For fabricating composites, calculated amounts of Ti$_3$AlC$_2$ (-325 mesh) or Cr$_2$AlC (-325 mesh), and Ni powders (Part number 43214 (-325 mesh and 99.8% pure) Alfa Aesar, Haverhill, MA) were mixed in a ball mill (8000 M mixer Mill, SPEX SamplePrep, Metuchen, NJ) for 5 minutes. The mixed powders were then cold-pressed at ~263 MPa in metal die (~12.7 mm diameter) then the samples were hot-pressed under a uniaxial compressive stress of ~240 MPa at 600 °C for 5 minutes. For comparison, Ni sample was also manufactured by the same route. This composition will be referred to as Ni (A-composition). In an earlier study, Ni samples were also synthesized under similar hot press conditions, but the compacts were cold pressed at ~88 MPa. This composition will be referred to as Ni (B-composition) [1]. The mass and dimensions of all the samples were measured to determine the experimental density ($\rho_E$). The theoretical density of the Ti$_3$AlC$_2$, Cr$_2$AlC and Ni particulates were used to calculate the theoretical density ($\rho_T$) of the composites by rule of mixtures. Thereby, the relative density of the composites were calculated by dividing the experimental density with the theoretical density of the samples. Porosity (P) of the sample was then determined by the Eq. 1.

$$P = \left(1 - \frac{\rho_E}{\rho_T}\right) \times 100$$

Eq. 1
The characterization techniques for mechanical testing by uniaxial compression, XRD for phase analysis, and hardness are mentioned in the Chapter II except tribology measurements. The tribology characterization was done by using SS balls as a tribo-partner in this by ball on disc method. All the other parameters are similar to Chapter II.

3.2 RESULTS AND DISCUSSION

3.2.1 MICROSTRUCTURE AND PHASE ANALYSIS

BSE SEM images of the MAX Phase powders Ti₃SiC₂, Ti₃AlC₂ and Cr₂AlC are shown in Figs. 1 a-c, respectively. The SEM micrographs of Ni-Cr₂AlC and Ni-Ti₃AlC₂ composites are shown in Fig. 2 and Fig. 3, respectively. All the compositions showed that MAX phase particulates are evenly distributed the Ni matrix. Moreover, no interfacial reactions were observed even at higher magnifications between Ni and Cr₂AlC (Fig. 2f) or Ti₃AlC₂ (Fig. 3f). XRD plots of these two composites are summarized in Fig. 4. All the compositions showed peaks of Ni and MAX particulates which further corroborates the SEM findings.

Figure 5 plots porosity versus the content of the MAX or MoAlB phase reinforcement in the Ni-Matrix. The Ni (A-composition) fabricated during study has ~9.82% porosity as compared to ~13% porosity in Ni (B-composition) [1]. This study shows that high cold pressing pressure is a critical requirement for manufacturing dense compositions by using the procedure mentioned above. Thus, the Ni-Ti₃AlC₂ and Ni-Cr₂AlC compositions fabricated during study are denser than Ni-MoAlB compositions fabricated during a previous study [1]. For example, Ni-5%Cr₂AlC, Ni-10%Cr₂AlC, and Ni-20%Cr₂AlC had similar porosities of ~10.7%, ~10.8%, and ~10.9%, respectively. The composition Ni-30%Cr₂AlC had a higher porosity of ~19.6% porosity. Similarly, Ni-5%Ti₃AlC₂, Ni-10%Ti₃AlC₂, and Ni-20% Ti₃AlC₂ had porosities of 9.8%, 11.8%, and 12.7%, respectively. Like the former case, the composition Ni-30%Ti₃AlC₂ had a higher porosity of
~19.6% porosity. Comparatively, Ni-5%Ti$_3$SiC$_2$, Ni-10%Ti$_3$SiC$_2$, Ni-20% Ti$_3$SiC$_2$ and Ni-30% Ti$_3$SiC$_2$ composites fabricated by similar method had porosities of ~11.8%, ~11.6%, ~12.5% and ~18.1%, respectively (Chapter 2). This study shows that the densification of Ni-MAX composites are showing a similar trend during manufacturing by using a combination of cold pressing and hot pressing.

Figure 5b shows the plot of hardness versus MAX or MoAlB content. Both the Ni compositions had similar hardness of ~1.6 GPa. For Ni-Ti$_3$AlC$_2$ compositions, the hardness decreased slightly from ~1.5 GPa in Ni-5%Ti$_3$AlC$_2$ to ~1.4 GPa in Ni-20%Ti$_3$AlC$_2$, respectively. The hardness of Ni-30%Ti$_3$AlC$_2$ composite decreased to ~1.1 GPa. For Ni-Cr$_2$AlC composites, the hardness retained a value of ~1.5 GPa until Ni-10%Cr$_2$AlC composite, thereafter it decreased to ~1.4 GPa in Ni-20%Cr$_2$AlC and ~1.1 GPa in Ni-30%Cr$_2$AlC composites. For comparison, Ni-Ti$_3$SiC$_2$ composites also showed a similar trend (Fig. 5b).

### 3.2.2 COMPARISON OF MECHANICAL PERFORMANCE OF MRMS

Figures 3.6a and 3.6b show the plots for the compressive stress versus displacement of the Ni-Ti$_3$AlC$_2$ and the Ni-Cr$_2$AlC composites, respectively. The addition of MAX phases gradually increased the brittle nature of the composites. Ni (Composition-A) had a yield strength of ~576 MPa, the yield strength increased to ~607 MPa in Ni-5%Cr$_2$AlC then decreased gradually to ~500 MPa in Ni-20%Cr$_2$AlC. Similarly, Ni-5%Ti$_3$AlC$_2$ had a yield strength of ~536 MPa and it decreased gradually to ~502 MPa in Ni-20%Ti$_3$AlC$_2$. At higher concentration, the yield strength of Ni-30%Ti$_3$AlC$_2$ and Ni-30%Cr$_2$AlC decreased sharply to ~247 MPa and ~384 MPa, respectively.
Figure 3.1: BSE SEM micrographs of, (a) Ti₃SiC₂, (b) Ti₃AlC₂, and (c) Cr₂AlC particles.
Figure 3.2: SE SEM image of, (a) Ni-5%Cr$_2$AlC, (b) Ni-10%Cr$_2$AlC, (c) Ni-20%Cr$_2$AlC, (d) BSE of the same region, (e) Ni-30%Cr$_2$AlC, and (f) BSE image of the highlighted region in (e).
Figure 3.3: SE SEM image of, (a) Ni-5% Ti$_3$AlC$_2$, (b) Ni-10% Ti$_3$AlC$_2$, (c) Ni-20% Ti$_3$AlC$_2$, (d) BSE of the same region, (e) BSE image of Ni-30% Ti$_3$AlC$_2$, and (f) BSE image of the highlighted region in (e).
Figure 3.4: XRD plot of, (a) Ni-Ti$_3$AlC$_2$, and (b) Ni-Cr$_2$AlC composites.
Figure 3.5: Plot of, (a) porosity, (b) hardness in Ni-matrix composites [1].

Figure 3.6: Plot of, compressive stress versus displacement in, (a) Ni-Ti$_3$AlC$_2$, (b) Ni-Cr$_2$AlC composites, and (c) yield strength versus MAX or MAB content (Ni-MoAlB data is from Ref. 1, and Ni-Ti$_3$SiC$_2$ data is from Chapter 2 of this thesis).
Figure 3.6: Plot of compressive stress versus displacement in, (a) Ni-Ti$_3$AlC$_2$, (b) Ni-Cr$_2$AlC composites, and (c) yield strength versus MAX or MAB content (Ni-MoAlB data is from Ref. 1, and Ni-Ti$_3$SiC$_2$ data is from Chapter 2 of this thesis).
3.2.3 SUMMARY OF TRIBOLOGICAL BEHAVIOR OF DIFFERENT MRMS

Figure 3.8a shows the $\mu_{\text{mean}}$ of the Ni matrix composites. The $\mu_{\text{mean}}$ of the Ni-Ti$_3$AlC$_2$ compositions showed an abrupt increase in the $\mu_{\text{mean}}$ from ~0.55 in Pure Ni (Composition B) to ~0.77 in Ni-5% Ti$_3$AlC$_2$, which increased to ~0.91 for Ni-10% Ti$_3$AlC$_2$. Thereafter, the $\mu_{\text{mean}}$ decreased to ~0.78 in Ni-30% Ti$_3$AlC$_2$. Comparatively, in Ni-Cr$_2$AlC composites, the samples had similar values of $\mu_{\text{mean}}$ of ~0.55 until Ni-10%Cr$_2$AlC, thereafter it increased to ~0.59 and ~0.73 in Ni-20%Cr$_2$AlC and Ni-30%Cr$_2$AlC, respectively. Comparatively, Ni-Ti$_3$SiC$_2$ and Ni-MoAlB also showed a similar trend where $\mu_{\text{mean}}$ increased with the concentration of these phases (Chapter 2 and Ref. 1). Qualitatively, if we compare all the Ni-matrix composites, Ni-Cr$_2$AlC composites showed lower $\mu_{\text{mean}}$ than all the tested composites.

The WR of the composites are plotted in Fig. 8b as a function of MAX or MoAlB content. Comparatively, Ni-Ti$_3$SiC$_2$ and Ni-Ti$_3$SiC$_2$ are showing similar trend where WR are in the regime of (2-4) x $10^{-3}$ mm$^3$/Nm. Comparatively, the WR of Ni-Cr$_2$AlC fluctuates from 3 x $10^{-3}$ mm$^3$/Nm in Ni (Composition-B) to 4 x $10^{-3}$ mm$^3$/Nm in Ni-20%Cr$_2$AlC, thereafter it decreased by 1 order of magnitude to ~4 x $10^{-4}$ mm3/Nm in Ni-30%Cr$_2$AlC. Comparatively, Ni-MoAlB compositions also showed similar trend where the WR decreased by 1 order of magnitude in Ni-30%MoAlB. This study clearly shows that Ni-30%Cr$_2$AlC and Ni-30%MoAlB compositions are resistant to adhesive wear.

3.2.4 STUDY OF TRIBOФILMS AND MECHANISM OF WEAR

Figures 3.8 and 3.9 show the tribosurfaces of Ni-30%Ti$_3$AlC$_2$ versus stainless steel and Ni-30%Cr$_2$AlC versus stainless steel tribocouples. If we inspect the Ni-30%Ti$_3$AlC$_2$ versus stainless steel tribocouples, then wear debris and trioxides can be observed on both the Ni-30%Ti$_3$AlC$_2$ (Figs. 3.8 a-b) and stainless steel (Figs. 3.8 c-d) tribosurfaces. Comparatively, N-30%Cr$_2$AlC (Fig.
3.9 a-b) and stainless steel (Fig. 3.9 c-d) surface was smooth and lubricous tribofilms was observed on both surfaces. Based on this work, we can conclude that Cr$_2$AlC is a more effective solid lubricant than other MAX phases or MoAlB. More studies are needed to understand the detailed mechanisms responsible for these results.

Figure 3.7: Plot of, (a) friction coefficient, and (b) WR versus MAX or MAB content in Ni-matrix composites.
Figure 3.8: SEM of tribosurfaces of, (a) Ni-30%Ti$_3$AlC$_2$ in SE, (b) BSE of the same region, (c) steel countersurface in SE, and (d) BSE of the same region.
Figure 3.9: SEM of tribosurfaces of, (a) Ni-30%Cr₂AlC in SE, (b) BSE of the same region, (c) steel countersurface in SE, and (d) BSE of the same region.

\[(\text{Ni}_{0.58}\text{Fe}_{0.29}\text{Cr}_{0.08}\text{Al}_{0.05})\text{O}_{0.78} \{\text{C}_x\}\]

\[(\text{Fe}_{0.98}\text{Cr}_{0.02})\text{O}_{0.02} \{\text{C}_x\}\]
CHAPTER IV
CONCLUSIONS AND FUTURE WORK

4.1 INTRODUCTION

Al-Si alloys have emerged as candidate materials for aerospace, automobile, and defense related applications, for example plain bearings, piston and cylinder liners in internal combustion engines etc. These alloys are bestowed with good thermal conductivity, mechanical behavior, and corrosion resistance [1-9]. Different metallic additives like Pb, Sn, Ti, and Cu are added to improve the mechanical and antiwear properties of AlSi alloys by engineering the microstructure [6-9]. Different researchers have also used ceramic additives like SiC, TiC and TiB₂ to enhance the mechanical and tribological behavior of these composites [10-12]. In this Chapter, we will report the tribological and mechanical behavior of AlSi-Ti₃SiC₂ composites.

4.2 EXPERIMENTAL DETAILS

Ti₃SiC₂ powder (-325 mesh, Kanthal, Hallstahammar, Sweden) and calculated amounts of Al-12wt%Si powders (Part number 88322 (-325 mesh and 99% pure), Alfa Aesar, Haverhill, MA) were initially dry mixed in a ball mill (8000 M mixer Mill, SPEX SamplePrep, Metuchen, NJ) for 5 minutes. The mixed powders were then hot-pressed under a uniaxial compressive stress of ~240 MPa (~12.7 mm die) at 500 °C for 10 minutes. Pure Al-12wt%Si samples were also fabricated at ~240 MPa by following procedure mentioned above. In this chapter, Al-12wt%Si composition will be referred to as AlSi. As a part of this study, ~5 vol% (Al-Si~5%312Si), ~10 vol% (Al-Si~10%312Si) and ~20 vol% (Al-Si~20%312Si) of Ti₃SiC₂ particulates were used as a reinforcement in the Al-Si alloy matrix.

The procedure for measuring porosity, mechanical behavior, hardness, and microstructure characterization by SEM is reported in Chapters II and III. In this study, the tribological study was
performed by block-on-disk tribometer (CSM Instruments SA, Peseux, Switzerland) method. The samples of AlSi-Ti$_3$SiC$_2$ compositions were cut into dimensions of ~4 mm (length) x ~4 mm (width) x ~3-4mm (height), and then polished to $R_a < 1 \mu m$. The polished samples were tested against polished alumina disks ($R_a < 3 \mu m$). The surface roughness of both the two surfaces were confirmed by using a surface profilometer (Surfcom 480A, Tokyo Seimitsu Co. Ltd., Japan). The experimental conditions of tribological testing were a load of 5 N, linear speed of ~50 cm/s, track radius of ~10 mm, and a sliding distance of 500 m. The mean friction coefficient was measured from each testing. For a particular composition, the mean friction coefficient from three measurements were averaged to calculate $\mu_{mean}$. The mass of the samples and substrates were measured before and after the testing by using a weighing scale ((Model XA82/220/2X, Radwag Balances and Scales, Poland). The specific wear rate (WR) was calculated from:

$$WR = \frac{(m_i - m_f)}{(\rho.N.d)}$$

where, $m_i$ is the initial mass, $m_f$ is the final mass, $\rho$ is density of the composite, N is the applied load, and $d$ is the total distance traversed by the sample during the tribology testing. In this text, the total WR from both the counterparts is reported. Please refer to Chapter II about the methodology for characterizing the microstructure and tribochemistry of the worn surfaces after tribology measurements.

4.3 RESULTS AND DISCUSSION

4.3.1 MICROSTRUCTURE AND PHASE ANALYSIS

Figure 1 shows the SEM images of all the fabricated Al-Si matrix composites along with the pure Al-Si sample. All the images show that Ti$_3$SiC$_2$ particulates are uniformly dispersed in the Al-Si matrix with minimal interfacial reactions (Figs. 4.1c and 4.1f). Figure 4.2 plots porosity (Y1 axis) and hardness (Y2 axis) of the composite as a function of Ti$_3$SiC$_2$ content. The porosity
of AlSi, Al-5%Ti₃SiC₂, and Al-10%Ti₃SiC₂ had similar porosity of 4.7%, 4.4%, and 4.8%, respectively. Comparatively, the porosity of Al-20%Ti₃SiC₂Al was comparatively higher at 7.9%. The hardness of the composites also gradually increased from 924 MPa in AlSi to ~982 MPa and ~994 MPa in Al-5%Ti₃SiC₂ and Al-10%Ti₃SiC₂, respectively. Thereafter, the hardness decreased to ~909 MPa in Al-30%Ti₃SiC₂.

Figure 4.1: BSE SEM micrographs of, (a) AlSi, (b) AlSi-5%Ti₃SiC₂, (b) higher magnification of the marked region in (b), (c) AlSi-10%Ti₃SiC₂, (d) AlSi-20%Ti₃SiC₂, (e) higher magnification of the marked region in (e).
Figure 4.2, Plot of porosity (%) (Y1 axis) and hardness (Y2 axis) as a function of Ti$_3$SiC$_2$ content.

4.3.2 COMPARISON OF MECHANICAL PERFORMANCE OF MRMS

Figure 4.3a plots the compressive stress versus displacement behavior of AlSi-Ti$_3$SiC$_2$ composites. The composites become more brittle as the content of Ti$_3$SiC$_2$ was increased in the AlSi matrix. Figure 4.3b plots the yield strength of AlSi-Ti$_3$SiC$_2$ composites as a function of Ti$_3$SiC$_2$ content. The yield strength increases from 263±26 MPa in AlSi to (283±56) MPa in Al-5%Ti$_3$SiC$_2$, thereafter it decreased to (211±106) MPa in Al-10%Ti$_3$SiC$_2$, and marginally increased to (230±94) MPa in Al-30%Ti$_3$SiC$_2$. 
Figure 4.3: Plot of, (a) compressive stress versus displacement, and (b) ultimate compressive stress (UCS) versus Ti$_3$SiC$_2$ content in AlSi matrix.
4.3.3 SUMMARY OF TRIBOLOGICAL BEHAVIOR OF DIFFERENT MRMS

Figure 4.4a shows the plot of $\mu_{\text{mean}}$ versus the amount of Ti$_3$SiC$_2$ added in the composite. In these composites, $\mu_{\text{mean}}$ increased from ~0.56 in AlSi-5% Ti$_3$SiC$_2$ to ~0.68 in AlSi-10% Ti$_3$SiC$_2$, thereafter it decreased gradually to ~0.59 in AlSi-20% Ti$_3$SiC$_2$.

Figure 4.4b shows the plot for the wear rate of the composites. The WR of AlSi decreased marginally from ~0.0005 mm$^3$/Nm in AlSi to 0.0003 mm$^3$/Nm in Al-5%Ti$_3$SiC$_2$ thereafter it increased to ~0.0004 mm$^3$/Nm and 0.001 mm$^3$/Nm in AlSi-10%Ti$_3$SiC$_2$ and AlSi-20%Ti$_3$SiC$_2$, respectively.

Figure 4.4: Plot of, (a) $\mu$, and (b) WR as a function of Ti$_3$SiC$_2$ content.
4.3.4 STUDY OF TRIBOFILMS AND MECHANISM OF WEAR

Figure 4.5: SEM micrographs of, (a) AlSi surface in SE, (b) BSE of the same region, (c) SE image of AlSi surface at high magnification, and (d) BSE of the same region (inset shows the BSE image of Al$_2$O$_3$ surface) during tribology testing.

Figures 4.5 a-d show the AlSi surface after tribological testing. The surfaces were riddled with abrasive wear tracks. Inset of Fig. 4.5d shows the alumina surface where the material transferred from AlSi surface is deposited. EDS results also show that the surface is partially oxidized. Figures 4.6 a-b and Figs. 4.6 c-d show AlSi-10%Ti$_3$SiC$_2$ and the corresponding alumina
surface after testing. It showed a similar trend like the previous case, and the material from AlSi-10%Ti₃SiC₂ surface was transferred to the alumina surface due to abrasive wear. The transferred material was also partially oxidized during the tribological testing.

![SEM micrographs](image)

Figure 4.6: SEM micrographs of, (a) AlSi-10%Ti₃SiC₂ surface in SE, (b) BSE of the same region, (c) SE image of the Al₂O₃ surface, and (d) BSE image of the same region during tribology testing.

### 4.4 CONCLUSIONS

Novel AlSi- Ti₃SiC₂ composites were fabricated by hot pressing. The hot pressed samples showed higher densities than most of the previously studied MRMs. The SEM images showed that the Ti₃SiC₂ particulates were well dispersed within the AlSi matrix with minimal interfacial reactions. The addition of Ti₃SiC₂ increased the yield strength from a mean strength of ~263 MPa.
for pristine AlSi to 283 MPa in AlSi-5% Ti$_3$SiC$_2$. Further addition of Ti$_3$SiC$_2$ decreased the yield strength to ~211 MPa for AlSi-10% Ti$_3$SiC$_2$, which marginally increased to ~230 MPa in AlSi-30% Ti$_3$SiC$_2$. The addition of Ti$_3$SiC$_2$ improved the tribological properties of the composite marginally. Detailed studies are needed to understand the fundamental mechanisms responsible for the observed tribological behavior.
REFERENCES

CHAPTER I


CHAPTER II


CHAPTER III


CHAPTER IV

CONCLUSION AND FUTURE WORK


