STUDIES ON TITANIUM NITRIDE REINFORCED ALUMINIUM METAL MATRIX COMPOSITES

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ABSTRACT

Aluminium (Al) reinforced with Titanium Nitride (TiN) Metal Matrix composites find its application for elevated temperature operating conditions. 5, 10 and 15 weight percentage of TiN particles was added to aluminium to prepare the composite through powder metallurgy technique. The mixed powder was compacted at two compacting pressure of 250 MPa and 300 MPa to produce the specimens having h/d ratio in the range of 1.1 to 1.2. The specimens were sintered in nitrogen atmosphere at two different sintering temperatures of 400°C and 500°C with sintering time of 4 hours for each. Physical properties namely, green density, sintered density and mechanical properties such as hardness, compression strength and surface roughness were studied.

Key words: Aluminium-TiN composites, compacting pressure, powder metallurgy, sintering temperature.


1. INTRODUCTION

Composites have established their suitability in the field of automotive and aerospace industries and research on metal matrix composites (MMCs) has shown tremendous promise in the recent past [1]. Particulate reinforced aluminium metal matrix composites display better physical, mechanical and tribological properties namely corrosion resistance, increased hardness, high specific strength and specific modulus, high wear resistance and low coefficient of thermal expansion over those of alloys [2-5].

The manufacturing processes like solid state (powder metallurgy) and liquid state (stir casting) have been developed to fabricate the particulate reinforced aluminium metal matrix composites in an effective manner [6]. Aluminium matrix composites fabricated via powder
metallurgy technique exhibit good isotropic properties and green density greater than 90% of theoretical density by using low compacting pressures between 200 to 250 MPa [7].

The most widely used reinforcement materials are metal carbides, oxides, nitrides and borides. SiC, Al2O3 and carbide particles are some of the commonly used reinforcement materials for aluminium. However, the use of SiC as reinforcement in aluminium matrix is limited due to its reaction with aluminium above 720°C resulting in formation of Al4C3, poor mechanical properties and low corrosion resistance [8].

TiN is a high melting point compound with extreme hardness, low electric resistance and good thermal stability [9-10]. It does not react with aluminium, because of its good stability up to a temperature of 3300 K. Also it is chemically inert to most of the acids. It is often used as coating on titanium alloys, steel and aluminium components to improve the substrate surface properties.

Aluminium-TiN composites under microwave sintering exhibit superior hardness and wear resistance properties as compared to Al-TiN composites developed by hot pressing. For proper sintering of specimens, conventional sintering takes a few hours while the same could be achieved in two minute using microwave sintering [11].

The presence of TiN particles at the grain boundaries in aluminium provides a better densification, improvement in mechanical and tribological properties. TiN is currently used in cutting tools, solar control films and other microelectronic application [12]. The present work therefore focuses on preparation of aluminium based composites containing 5, 10 and 15 weight percentage of TiN particles through powder metallurgy route. An attempt has also been made to study the influence of compacting pressure and sintering temperature on physical and mechanical properties of composites.

2. MATERIAL AND EXPERIMENTAL METHODS

2.1. Materials

![SEM images of Al and TiN powders](image)

**Figure 1** Scanning electron micrograph of as received (a) Al powder and (b) TiN powder.

Atomized Aluminium (Al) powder (99.5 % pure) and Titanium Nitride (TiN) as supplied by Metal Powder Company Limited, India and Sigma Aldrich, Germany was used as matrix and reinforcement materials respectively. Figure 1 (a) and (b) shows the SEM images of Al and TiN powders The specifications of Al and TiN powder are shown in Table 1 and Table 2 respectively.
Table 1 Specification of aluminium powder

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Particle Size</th>
<th>Atomic weight</th>
<th>Arsenic</th>
<th>Lead</th>
<th>Iron</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specification</td>
<td>74 µm</td>
<td>26.98</td>
<td>0.0005%</td>
<td>0.03%</td>
<td>0.5%</td>
</tr>
</tbody>
</table>

Table 2 Specification of titanium nitride powder

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Particle Size</th>
<th>Molecular weight</th>
<th>Melting point</th>
<th>Density</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specification</td>
<td>&lt;3µm</td>
<td>61.87 g/mol</td>
<td>29300°C</td>
<td>5.24 g/cc</td>
</tr>
</tbody>
</table>

2.2. Preparation of Composites

Initially, TiN powder in different weight percentages 5, 10 and 15 were mixed with aluminium using horizontal mill for about 30 minutes. During mixing a control agent was added to avoid agglomeration and cold welding of powder particles. Each of the blended mixtures were pressed at two different compaction pressures of 250 MPa and 300 MPa using uniaxial hydraulic press and lubricated dies.

The cold pressed specimens were sintered in nitrogen atmosphere at 400°C and 450°C for 4 hours using tube furnace. Figure 2 depicts sintered samples of Al-TiN.

Figure 2 Samples of compacted and sintered Al-TiN composite

The density of compacts were measured by volumetric method, where weight and dimensions were measured with the help of an accurate weighing balance (+0.01mg) and a micrometer (+0.1 mm) respectively. Brinell hardness of the samples was measured. Before the compression test, the surfaces of the specimen were polished using an emery paper to have uniform distribution of load. Compression test was conducted according to ASTM-D 618. The surface roughness of each specimen was determined using surface roughness tester and an SEM micrograph was used to study the morphology of sintered specimens.

3. RESULTS AND DISCUSSION

3.1. Density of Composites

Composite specimens were prepared under two different compacting pressures of 250 and 300MPa to be indicated as P1 and P2 respectively. The sintering temperature used for the specimens was 400°C and 450°C to be referred as T1 and T2 respectively. It was found that the average green (un-sintered) density and sintered density of composite specimens increase with increase in weight percentage of TiN. Figure 3 and Figure 4 depicts the variation in green density of Al-TiN composites in pressed and sintered condition of specimens.
It is seen that the theoretical density of composites increase with increase in weight % of TiN as the density of TiN is greater than that of aluminium. The measured density does not show similar trend to that of theoretical density due to varying porosity. The comparison between the theoretical and measured density of Al-TiN composites are shown in Figure 5. The sintered density of compact was observed to be lower than that of green ones as during sintering the mixing aid was lost leading to enhanced porosity. The results indicate that measured densities vary between 94 to 98 % of theoretical density.
3.2. Hardness

Three set compacts were used to determine Brinell hardness number of Al-TiN composites for each weight percentage of TiN. Figure 6 shows the variation of hardness of the Al-TiN composites for different weight percentage of TiN.

![Figure 6 Variation of Hardness for different weight percentages of TiN reinforced Al composites at two different compacting pressure and sintering temperature](image)

The hardness of composite with 10% TiN was found to be 17% higher than the unreinforced aluminium sample. It was observed that Al-TiN composite pressed at 300 MPa, followed by sintering at a temperature of 450°C for a sintering of 4 hours exhibit higher hardness.

3.3. Compression Strength

Figure 7 shows the compression strength of Al-TiN composite specimens. The findings indicate a significant increase in compression strength with the addition of TiN reinforcement into aluminium. This is due to the reinforcing effect on aluminium. Uniform distribution of particles has resulted in improved compression strength. The compression strength of 15 wt%
TiN reinforced aluminium composite was found to be 37% higher than that of unreinforced aluminium.

![Figure 7](image_url) Variation of Compressive strength for different weight percentages of TiN reinforced Al composites at various compacting pressure and sintering temperature

### 3.4. Microstructural Analysis

The micrograph of the Al-TiN composites was investigated by SEM and respective micrographs of the compact taken at their cross-section are shown in Figure 8. The amount of porosity, pore size, shape and distribution within the samples were investigated using SEM equipped with EDAX analysis.

The micrographs indicate that there is a uniform distribution of TiN particles within the matrix. The composite with 5 wt% (Figure 8b) shows the presence of TiN particles (bright area) uniformly throughout the Al matrix (dark areas). As the content of reinforcement increases from 5 to 15 wt%, there is an improvement in overall distribution of these particles in the matrix. The micrograph of the composites reveals the presence of porosity, since the sintered density of composite is between 85 to 95% of theoretical density. Medium size pores and small interconnected micro pores were observed at grain boundaries.

Figure 9 and Table 3 depicts the elemental microanalysis of matrix and of various grain boundaries obtained using EDS analysis. It is observed that, the structure contains basically Al, Ti and N. However, it can be seen O is present due to high affinity of Al in the ambient temperature. The EDAX shows that, the presence of grain boundary was almost consistent in all the sintered samples.
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![Figure 8](image)

**Figure 8** Scanning electron micrograph of (a) pure Al and composites reinforced with TiN particles having (b) 5, (c) 10 and (d) 15 wt% TiN particles in pure Al matrix

![Figure 9](image)

**Figure 9** SEM micrograph and EDAX analysis of Al- 5Wt % TiN

**Table 3** EDAX analysis of Al -5 Wt % TiN compact.

<table>
<thead>
<tr>
<th>Elements</th>
<th>N</th>
<th>O</th>
<th>Al</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight %</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.55</td>
<td>5.59</td>
<td>87.93</td>
<td>4.93</td>
</tr>
</tbody>
</table>

3.5. Surface Roughness

Figure 10 depicts the variation of surface roughness of the Al-TiN composites. The surface roughness of samples varies from 5 µm to 1.43 µm. The surface roughness is minimum for the sample containing 15% of TiN reinforcement, which is because of higher cold working and recrystalization. Results indicate as higher compacting pressure and sintering temperature lead to reduction in the roughness of the surface.
4. CONCLUSION
1. Mixing of aluminium and TiN powders in a horizontal mill for 30 minutes resulted in good compacts.
2. Uniaxial compaction at 250 MPa and 300 MPa followed by sintering in a nitrogen atmosphere at 400°C and 450°C has been used successfully to develop Al-TiN composites.
3. Brinell hardness, density and compressive strength of composites increase with increase in reinforcement content from 5 to 15 weight percent of TiN.
4. The surface roughness of the composites developed decreased with increase in the amount of reinforcement, compacting pressure and sintering temperature.

REFERENCES
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