

## Effect of Ageing and Amount of Tic on Hardness of Al/Tic Composites

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**ABSTRACT:** This paper describes about influence of both ageing response and amount of TiC reinforcement on Al composites properties such as age hardening, electric resistivity and microstructure changes. The samples were aged at 30, 100, 170, and 240°C for 0.01 to 100 hours. The peak of the hardness and resistivity values of the MMCs were increased with increasing TiC content and ageing temperature. Ageing acceleration precipitation was observed more significantly in higher percentage reinforced composite due to higher dislocation density. Dislocation density helped to nucleate the precipitation, which is higher in composites due to mismatching thermal and mechanical properties between reinforcement and matrix. Changes in the properties of the composites during ageing have been explained on the basis of micro structural alterations during ageing.

**Keywords:** Ageing, Al/TiC composites, hardness, DSC.

### I. Introduction

Aluminium composites generally called precipitation hardenable matrices and when ceramic particle reinforced the ageing considerably faster than matrix alloy [1]. The formation of dislocation density in the composites is higher than that of unreinforced matrix alloy due to thermal mismatch between the reinforcement and matrix alloy. This dislocation helps the diffusion of solute atoms which leads to formation of nucleation sites which leads to rapid precipitation process [2]. The quenching temperature and solution temperature play significant role in the decomposition and formation of precipitation between the alloying elements [3]. The faster rate quenching rate enhances the Guinier Preston (GP) zone formation along with some intermediate Al, Si and Mg based precipitations [4]. Transmission electron microscopy (TEM) studies have the heterogeneous nucleation formation in the matrix alloy improves the precipitation rate which leads faster ageing kinetics [5]. Many researchers worked on the theoretical analysis of ageing kinetics in composites with some experimental results have recommended that the dislocation density of matrix alloy also influence the precipitation with shorter path for solute diffusivity [6].

The purpose of the research work was to investigate the effect of ageing temperature and TiC on the microstructure and hardness properties of TiC reinforced Al composites. The variation of hardness of composites was studied as a function of ageing time, temperature and TiC content. Energy dispersive X-ray spectra-meter and differential scanning calorimetry (DSC), and TEM investigation have been studied to investigate the effect of TiC on the decomposition and precipitation kinetics of the matrix alloy.

### II. Experimentation

Al 6061 alloy was used as the matrix material due to its good casting and strength to weight ratio. This alloy generally used for domestic application due its mass production and corrosion resistance, the chemical composition of Al given in Table 1.

Mg	Si	Cu	Mn	Al
0.92	0.76	0.22	0.04	Bal.

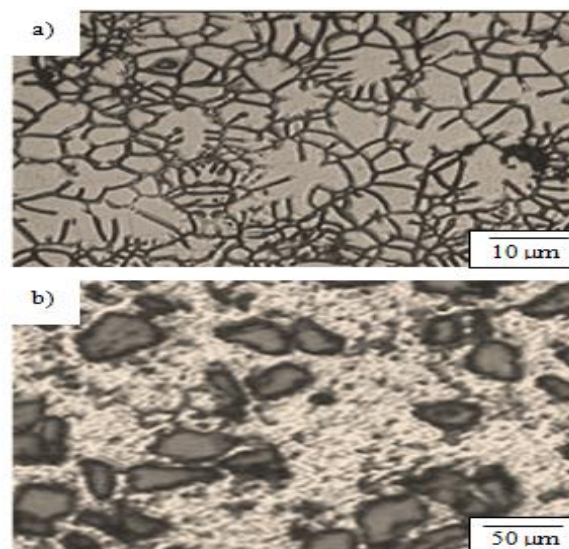
The TiC powder size of 1-3 µm was selected as the reinforcement and loading of TiC into composites varies 0, 5%, 10% and 15% by weight. Only 15% of TiC is restricted due to settling the reinforcement during mixing for higher percentage of reinforcements. Stirring technique was used for fabrication of TiC composites specimen. The preheated Nickel coated TiC particles were mixed with molten metal during stirring around 500 rpm of molten metal. The molten liquid degassed by nitrogen gas for 5 min then poured into preheated suitable die.

The heat treatment or ageing was done after specimen preparation as per standard. the specimen initially placed in the furnace for homogenous heat treatment for 24 hours at temperature of 523 °C then quenched it to ice cooled water for one hours then placed for natural ageing under room temperature for 24 hours and finally exposed to different ageing temperature at 30, 100, 170 and 240 °C for different time intervals from 0.01 to 100 hours.

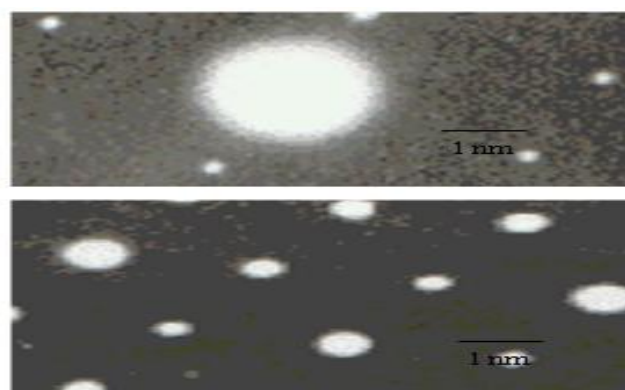
The cleaned and polished specimens were etched for optical micrography was done on the samples using Keller's reagents [7]. The polished specimens were used for microhardness test, which were conducted using a Leitz Wetzlar microhardness tester with the load applied was 1 N. Six hardness values were taken for each specimen and average values are drawn in the graphs. Care was taken to avoid making an indentation directly on a reinforcing particle which otherwise would cause a great scatter in the hardness value.

DSC used for measuring the total heat flow as a function of temperature for temperature range from 30 to 600 °C. The rate of heating was 10 K/ min. and the reference sample was indium. The phase transformations during ageing were analyzed by EDS,

TEM was used to investigate the dislocation density generated during heat treatment of Al/TiC composites. The composites specimens for TEM the thin 100 µm foils were fabricated using grinding followed by twin jet polishing with methanol and nitric acid mixture at -50 °C in the HNO<sub>3</sub> solution until the hole formed.



**Fig. 1** Microstructure of a) Al6061 alloy and b) Al/10% TiC composites



**Fig. 2.** Higher dislocation around TiC particle in composites (TEM) and b) precipitates in matrix after ageing at 180 °C for 10 hours (TEM).

### III. Results

#### 3.1. Microstructure

Microstructural properties of composites plays significant role in the performance (physical and mechanical properties) of matrix alloy and its composites. The mechanical properties mainly depend on reinforcement shape, size and distribution in the matrix materials. Fig. 1(a) and (b) show microstructure of Al matrix alloy and Al/10%TiC composites respectively. Fig. 1 (b) shows that uniform distribution of TiC particle in the matrix alloy. The size of the grain boundaries of Al matrix alloy is larger than that of Al/TiC composites. But some places in microstructure few large TiC particle cluster seen and entirely TiC depleted. The particles are restricted the dendrites grow within the matrix alloy due to solute enrichment and solidifies eutectic liquid around the TiC reinforcement. There is no inter-particle space is observed at the interface between the reinforcement and alloy matrix.

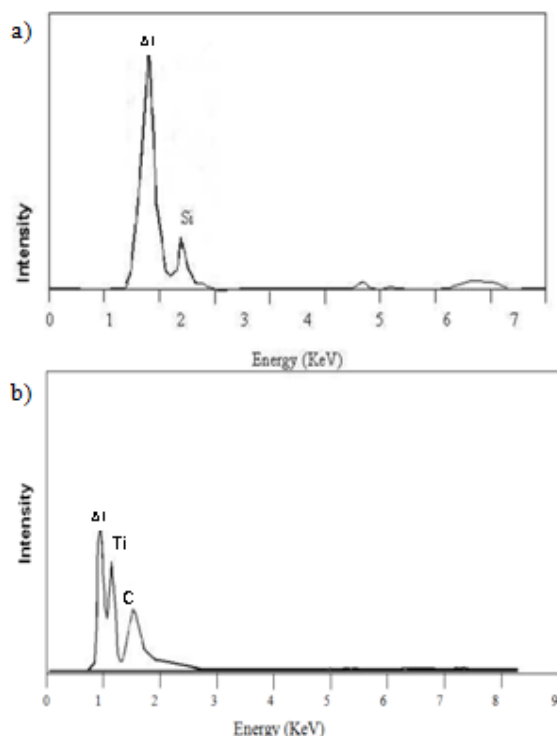


Fig. 3 EDS spectrum of the a) Al matrix alloy and b) Al/TiC composites respectively

When magnesium and Si elements present in the aluminum alloy, magnesium combined with  $\text{Al}_2\text{O}_3$  and forms  $\text{MgAl}_2\text{O}_4$  and  $\text{MgO}$ [8], the  $\text{Al}_2\text{O}_3$  is formed during stirring of molten metal for preparing composite [14]. Fig 2(a and b) shows the bright-field (TEM) of the aged Al/TiC composite for 10 hours. The higher density precipitates are observed in the TEM image and it is identified as silicon clusters. The  $\text{Mg}_2\text{Si}$  precipitates of size 20 nm are formed along grain boundaries and surrounding reinforcements they are generally called Guiner-preston zones as shown in Fig. 2(b).

Few researchers [9-10] reported that small precipitates formed along the interface between the reinforcement and matrix alloy their chemical compositions are  $\text{MgO}$  and  $\text{Al}_2\text{O}_3$  spinal particles.

At higher temperature (180 °C) formation of GP zone very small and needle shaped precipitates. The EDS revealed that the composition of aged composites specimen consists of  $\beta$ ,  $\text{MgO}$ , and  $\text{Al}_2\text{O}_3$  shown in Fig.3 (a and b).

#### 3.2. Microhardness results

The variations of microhardness ageing duration and temperature for both matrix and composites are shown in Fig. 4. The plots indicate that in all the cases except at an ageing temperature of 593 K, the hardness value initially ageing time sharply increment but after a peak value the hardness value drastically decreased. The addition of TiC in composites enhances the hardness of the composites due to formation of dislocation density between the reinforcement and matrix. The hardness values of different % TiC addition do not show any variation at longer duration of ageing due to dissolution of dislocation density within the materials. But the

composites overage (240 °C) formation of dislocation density is less pronounced. The over ageing influence the faster accelerated precipitate hence the maximum hardness is observed at early stage only.

At higher ageing temperatures, the precipitation and transformation of new phases occur very rapidly as the diffusion rate in solid solutions increases with increasing temperature. This explains the shifting of the hardness peak towards lower ageing time as the ageing temperature increases. At a higher ageing temperature of 170°C, peak is not observed since it must have been already achieved by the time the first observation was recorded.

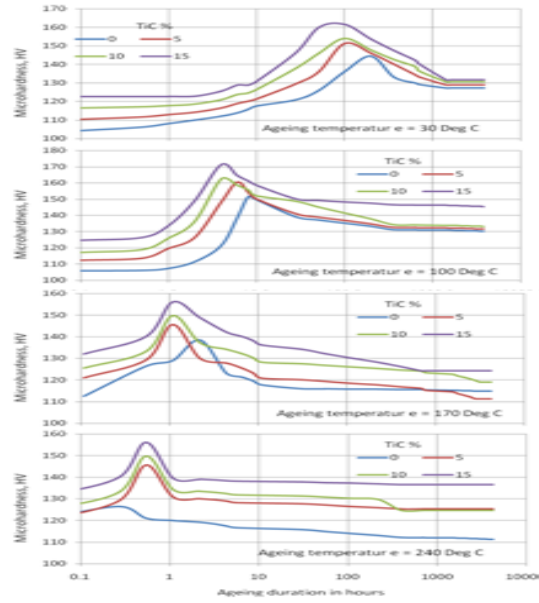


Fig 4 Microhardness curves of Al alloy and Al/TiC composites obtained on ageing at various temperatures.

### 3.3. DSC studies

The DSC results shows that different exothermic and endothermic peaks for both unreinforced and composites are shown in Fig. 5. The peak precipitation temperatures for composites significantly decrease with addition of TiC particles. It explains the TiC enhances the precipitation formation for different phases. The size of peaks precipitation curves are directly proportional to the weight % of TiC particles.

Previous works [11-12] on Al alloys have provided some evidences the formation of precipitation and rate of precipitation increasing with increasing, quenching and solution temperature. The shape of the precipitation at higher solution temperature is needle shaped structure and formed at interface between the particles.

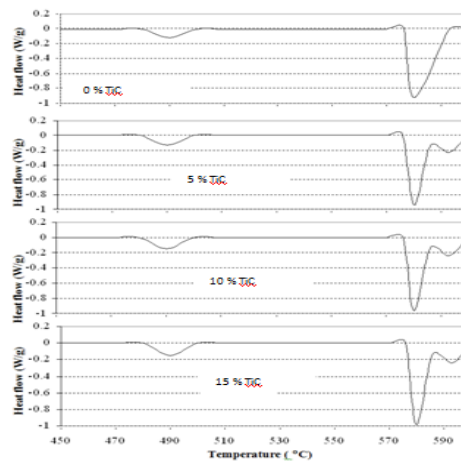


Fig. 5. The result of the differential scanning calorimetric for the Al alloy and its composites.

#### IV. Conclusion

1. On quenching Al alloys into cold water at 0°C after heat treatment, both precipitation and cellular reaction has been found to occur.
2. The hardness of the both matrix alloy and composites are increased with ageing duration up to peak then it decreased with time due to formation of precipitation at initial stage and dislocation of precipitation after the peak properties.
3. The similar trend was observed for DSC of both matrix and composites due to formation of dislocation density along the grain boundaries and nucleation concentrations.
4. The addition of TiC increases the hardness and also accelerates the ageing time.

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