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Analysis of thermo-elastic and physical properties of recycled 2017 Aluminium Alloy/Gp composites: thermal management application

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Abstract
This paper investigates the use of graphite in 2017 Aluminium matrix composite for thermal management systems by analyzing their thermo-elastic behavior. The composite samples were prepared with different graphite contents using powder metallurgy method based on recycled 2017 Aluminium alloy powder. The distribution of graphite particles in the 2017 Aluminium alloy matrix was investigated by a scanning electron microscope (SEM). Sintered densities of the composite samples were examined by a gravimetric method and it is noticed that in the fact of increasing the graphite content decreases the densification of composites which is confirmed by SEM analysis. Thereafter, the obtained samples were analyzed by dilatometry through measuring the coefficient of thermal expansion (CTE), which showed a decrease with the addition of graphite.

1. Introduction

Thermal management of electronic devices has become a serious concern for high performance applications [1]. This application need to provide high thermal conductivity (TC) and suitable coefficient of thermal expansion (CTE). Desirable metals for the electronic packaging materials are Aluminum (Al) and Copper (Cu) alloys. These have not matched thermal dissipation requirements due to their higher thermal expansion. In fact, temperature variations can induce thermal stresses due to the coefficient of thermal expansion (CTE) mismatch between devices which provoke electronic failures when the load is repeated. To withstand these extreme operating conditions, low and reasonable CTE with high thermal conductivity, can be achieved by blending appropriate phases to form a composite. Zweben [2] investigated the possibility of using particle reinforced metal matrix composites (PMMC’s) instead of monolithic metals. Since they offer significant improvements and distinct advantages in terms of their high TC carried by the Aluminium alloy matrix to dissipate the heat, low CTE carried by the particles in order to reduce the thermal stresses and the ability to be processed using a technology similar to that used for monolithic materials [2–4]. Reinforcements of Aluminium alloy matrix composites with high thermal conductivities are SiC, graphite or diamond. Graphite particles are good proposals [4–8] for thermal management compared to SiC and diamond [9–12].

Among the developed PMMC’s, composites made of recycled Aluminium alloy reinforced with graphite particles would be good proposals for thermal management.

In addition, to improve the strength and ductility of recycled Aluminium alloy, it has been suggested to refine its internal structure until powder scale. Nevertheless, a widespread use of the resulting composites would require a deep understanding of their TC and CTE and an optimization of the graphite content in the composite.

On the other hand, the amount of chips fabricated during the manufacturing of the final products of Al is huge. This can be prohibited by using recycling method required for industry [13, 14]. It has been suggested to produce secondary Al in order to save primary Al, by using recycling methods. The principal advantages of recycling Al are among many environmental benefits and low production cost.

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Aluminum chips can often be recycled by conventional methods involving both the re-melting and the atomization of re-melting chips processes in order to obtain powder. However, this procedure is complex due to the elongated spiral shape, and the small size of the chips [15]. Furthermore, new oxide skin formed during the re-melting process have an obvious effect on metal losses. About 48% of chips mass is lost during this process [16–19]. Additional disadvantage of this method is the high cost required due to the large number of steps to get a powder as centrifugal atomization [20], water atomization [21], chemical vapor deposition (CVD) [22] or electrolysis [23]. In addition, the high pollution is mainly due to the fumes and dross generated during the process [16, 17, 24].

To avoid these troubles, direct conversion method based on powder metallurgy technique have been developed [15, 17, 24, 25] to produce powder in a single step. Many authors [15–17, 24, 26, 27] confirmed that the latter one is more appropriate in terms of energy consumption (26%–31%) and materials saving (up to 40% of material) [16–19].

By adding extrusion process, this method has been used to produce, in particular, composites based on Al and AlCu4 alloy chips and tungsten powder and on 7075 Aluminium alloy chips mixed with Al powder are investigated in [24, 25] respectively. This methodology permits to have less mechanical properties than those of metallurgically-produced Al.

Including mechanical milling based on powder metallurgy technique [28–30] in the direct conversion provides powders with superior properties [31] compared to powder obtained by methods mentioned above. According to this technique, J. Gronostajski et al [15] approved that the use of powder as the initial material can offer important advantages including lower porosity, stronger mechanical properties and better control of the microstructure. In order to produce a denser structure at the best condition, it is necessary to study the effect of powder metallurgy parameters on the microstructure and physico-mechanical properties. This was the aim of our previous study [32] where the recycling of 2017 Aluminium alloy using this economical method has been treated for the first time.

As mentioned above, this recycled powder reinforced with graphite particles could be used in electronics and thermal management. Thereby, CTE are used as the primary parameter for this application, since it gives information about how much stress there is in electronic components. Thus, the goal of this study is design 2017 Aluminium alloy matrix composites with suitable CTE. These composites will have the potential to replace current electronic packaging material and improve the durability for thermal management of electronics.

The present investigation aims primarily to produce a denser samples by direct conversion based only on powder metallurgy. The obtained powder was then mixed with graphite particles, compacted and sintered in order to form composites. Secondly, to establish the effect of graphite content on the 2017 Aluminium alloy matrix composites through the density measurement and series of experiments by using a scanning electron microscope (SEM). Then, the effect of sintering parameters on the density and microstructure of 2017 Aluminium alloy was analyzed. Finally, it aims at studying the effect of graphite content on the thermal expansion behavior of the 2017 Aluminium alloy matrix composites. In this case, CTE of composites were measured by dilatometry.

### 2. Experimental procedure

The 2017 Aluminium alloy rods purchased from Sapa Profili Srl Company with the chemical composition listed in table 1 are used in the present study.

Figure 1 shows a schematic view of the synthesis process based on the direct conversion of the 2017 Aluminium alloy chips and the powder metallurgy method to fabricate composites.

| Element | Al | Si | Fe | Cu | Mn | Mg | Cr | Ni | Zn | Ti | Pb | Zr |
|---------|----|----|----|----|----|----|----|----|----|----|----|----|
| Mass %  |    |    |    |    |    |    |    |    |    |    |    |    |
| Bal.    | 0.66 | 0.27 | 4.01 | 0.64 | 0.57 | 0.07 | 0.01 | 0.01 | 0.03 | 0.04 | 0.12 |    |

The 2017 Aluminium alloy rods are machined using dry turning process in order to obtain a clean chip. The cutting conditions of speed and feed rate adopted during the machining operation were 200 m min\(^{-1}\), 0.2 mm tr\(^{-1}\), respectively. To get a finer thickness of chips, the cutting depth value was about 0.25 mm. The produced chip is found with curled forms, as shown in figure 1(a), with final dimensions: a length between 20 and 75 mm, a width less than 1 mm, and a thickness of 0.25 mm.

The obtained chips were transformed into small and regular particles. Then, 40 g of those chips were mechanically milled through a high-energy ball milling process for 20 h with a ball-to-powder weight ratio (B/P) of 10:1 at 250 rpm. In order to reduce excessive cold welding of the particles, 3 ml of methanol was affixed.
to powder. In our previous paper [32], the optimum milling time (20 h) has been approved to have a finer powder (figure 1(b)). Figure 1(c) displays a typical scanning electron microscope (SEM) image of the obtained 2017 Aluminium alloy after recycling and milling step [32] used in this work with an average particle size of 90 μm.

Timrex (SFG75) powder supplied by Timcal Graphite & Carbon (figure 1(d)) with spherical shape and an average size of 75 μm [33, 34] were used as the reinforcement component to produce composites. The 2017 Aluminium alloy powder and Graphite powder were mixed using planetary ball mill for 30 min. Three types of mixtures with different graphite content (0 wt%, 5 wt% and 10 wt%) were prepared under the same conditions used for 2017 Aluminium alloy (figure 1(e)).

Then, 80 g of composite powder was poured into the die cavity (85 × 65x10 mm³) and placed between two heating plates. Care was taken to ensure that the powder was properly dispersed in the die cavity. Thereafter, hot press process was executed on a hydrostatic press 'JOOS' of 1000 kN maximal pressing force (figure 2) to produce compacted powder at 200 °C and under a constant pressure of 150 MPa for 2 h. According to the results presented previously by Bhouri and Mzali [32], using these parameters allows the formation of denser structure of composites.

Hot-pressed samples with the dimensions 40 × 30x5 mm³ were shredded and sintered in a muffle furnace 'LabTech' at a heating rate of 20 °C min⁻¹, then cooled to room temperature (figure 3). Different times (t) and temperatures (T) combinations were used as illustrated in table 2 to investigate their effect. Figure 1(f) illustrates the sintered composite samples after polishing step.

Microstructure analysis were carried out using Scanning Electron Microscope (SEM) 'Thermo Fisher Scientific' to investigate the effect of different combinations during sintering and graphite particles on the microstructure of 2017 Aluminium alloy matrix composite. For this analysis, the SEM composites samples were prepared through grinding with 1200 SiC papers followed by polishing with Al₂O₃ suspensions to obtain mirror-like surfaces.

The green densities of sintered composite samples (ρₛ) were measured based on the formula (equation (1)):

\[ \rho_s = \frac{m}{V} \]  

where m and V are the mass and volume of each composite sample, respectively. The results were averaged over three independent measurements.

The volume percent of porosity was measured by comparing the theoretical density determined by rule of mixture and sintered densities (equation (2)):
where $P$ is the porosity and $\rho_{th}$ is the theoretical density of composite.

The theoretical densities of the composites were determined using the rule of mixture (equation (3)), in which the theoretical densities of 2017 Aluminium alloy and graphite were 2790 Kg m$^{-3}$ [35] and 2240 Kg m$^{-3}$ [33, 34], respectively.

$$\rho_{th} = \rho_{m} V_{m} + \rho_{p} (1 - V_{m})$$  \hspace{1cm} (3)$$

Where $V_{m}$ and $\rho_{m}$ the volume fraction and the density for the matrix ($\xi = m$) and for the particle ($\xi = p$).

The coefficient of thermal expansion was measured by using the dilatometer (TMA SETSYS Evolution) in the temperature range from 42 °C to 500 °C (figure 4). Composite samples with $8 \times 8 \times 4$ mm$^3$ were used. The

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**Figure 2.** (a) Schematic illustrating device of hot compaction technique and (b) Hydrostatic press 'JOOS'.

**Figure 3.** Schematic illustrating sintering cycle.

**Table 2.** Combinations of time and temperature during the sintering process.

| Combination (time, temperature) | Value                  |
|---------------------------------|------------------------|
| $(t_1, T_1)$                    | (45 min, 450 °C)       |
| $(t_1, T_2)$                    | (45 min, 550 °C)       |
| $(t_2, T_1)$                    | (90 min, 450 °C)       |
| $(t_2, T_2)$                    | (90 min, 550 °C)       |
top and the bottom of each sample were polished to guarantee plane–parallel surfaces for the measuring probe. In each test, the sample was positioned vertically and a moveable probe was placed on the top of the sample with a negligible force. The thermal expansion of the specimens was detected by a linear position transducer during heating and cooling controlled to 10 °C min\(^{-1}\) and 30 °C min\(^{-1}\), respectively. The heating and cooling rates during the measurements were continuously monitored by the computer based data acquisition system. The furnace surrounded both the sample stage and the probe under the protective argon gas atmosphere. The sample temperature was measured using a thermocouple, positioned close to the sample.

A typical curve showing a displacement–temperature hysteresis under thermal cycling is shown in [36, 37]. This hysteresis curve is useful to indicate the deformation of the material; its expansion and contraction in response to fluctuations in the operating temperature. It was quantified by exploring the parameter \( \Delta \varepsilon \) (equation (4)) which is defined as the strain range. Also, \( \varepsilon \) (equation (5)) is considered as the residual plastic strain [36, 37]:

\[
\Delta \varepsilon = \frac{\Delta L}{L_0} \quad (4)
\]

\[
\varepsilon = \frac{\Delta L}{L_0} \quad (5)
\]

These parameters were determined for different content of graphite particles and then presented in table 3.

Hysteresis curves were also used to calculate the CTE of the composites. The following equation allows to calculate the CTE from the measured dimensional change (\( \Delta L \)) over a temperature interval (\( \Delta T \)), where \( L_0 \) is the initial length of the sample:
Due to furnace fluctuations at temperatures lower than 250 °C which caused inaccuracies in the determination of CTE values, only CTE data above 250 °C were reported.

3. Results and discussion

3.1. Density—porosity of sintered composite

Figure 5 shows both density and porosity of 2017 Aluminium alloy matrix composites samples as function of graphite content, measured for different sintering time and temperature conditions previously presented in table 2.

A gradual decrease in composite density upon increasing graphite content is depicted on figure 5(a). This can be attributed to the lower density of graphite compared to the 2017 Aluminium alloy. Furthermore, the addition of graphite particles reduce the contact area between 2017 Aluminium alloy particles which provides a poor network and a reduction of the sintered density agreeing with many researches [4, 38–40]. Also, by exploring the measured densities of composites as function of graphite content for different sintering time and temperature conditions and according to the equation (2), the porosity of composites is calculated and plotted in figure 5(b).

It can clearly be seen that with the addition of graphite particles, the porosity increases drastically. In fact, the addition of Graphite during powder metallurgy method evolves gases that may be entrapped in the interior of the microstructure causing high amount of porosity [41].

\[
CTE = \frac{1}{L_0} \left( \frac{\Delta L}{\Delta T} \right)
\]
The figure 5(a) shows as well that the density gradually increases by increasing the sintering couples (table 2), thus the formation of a denser structure. At the same sintering time, the temperature has marginal effect on the densification of these samples. The figure 5(a) proves that the increase of sintering temperature from $T_1 = 450 \, ^{\circ}C$ to $T_2 = 550 \, ^{\circ}C$ leads to the increase of the sintered density. This contributes to the diminishment of the porosity. For instance, it is evident from the figure 5(b) that the porosity value after sintering at $550 \, ^{\circ}C$ is only 16.4%, compared to 19% obtained after $450 \, ^{\circ}C$.

This conclusion is justified by the diffusion phenomenon which is thermally activated according to many authors [4, 39, 40, 42, 43]. In this regard, they explained that the best particle bonding is taking place at a high temperature, and consequently, the pore reduction has a direct relationship with the sintering process.

Another parameter which improves the rise of the sintered powder density is the sintering time. It can be seen from figure 5(b) the good correlation between sintering time and porosity reduction. Indeed, with the increase of sintering time, the sintered density increases, and subsequently the porosity values decreases, as shown in figure 5(b). This was attributed to the ability of the sintering time to enhance diffusion, then allowing for increased grain and grain boundary development, as well as a reduction of porosity which is prominent during long stages of sintering process. Enhanced densification with the increase of sintering time was consistent with the work of Mehdi et al [44] wherein the dependence of the diffusion to time has been shown.

For example, for the same graphite amount (5 wt%) and sintering temperature ($550 \, ^{\circ}C$), the porosity of composite decreases from 16.4% to 13%, with increasing of sintering time (45 min to 90 min). The mechanism diffusion rate is activated not only by increasing the sintering temperature but by the increase of sintering time [44, 45].

Therefore, a higher densification is achieved at higher sintering time and temperature (90 min, $550 \, ^{\circ}C$).

3.2. Microstructure analysis
To prove the effect of Graphite particles on the sintered density of composites, microstructures of different Composite samples with lowest and highest sintering parameters combinations and Graphite content are analyzed by SEM as shown in figures 6 and 7.

Figure 6. SEM of microstructures showing the polished surface of sintered 2017 Aluminium alloy powder sintered at (a) $t_1 \, T_1$ and (b) $t_2 \, T_2$. 

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In the figure 6(a), a high porosity is visible, however, this porosity is reduced by the increase of sintering time and temperature as shown in figure 6(b). This indicates that the sintering combinations is predominant owing to a rise of the sintered density of 2017 Aluminium alloy matrix. This change is justified by the creation of necks between particles at (t1, T1). These necks are more developed for increased sintering parameters (t2, T2) (figure 6(b)), consequently, the formation of denser microstructure of 2017 Aluminium alloy matrix is achieved. This is consistent with density measurement (figure 5(a)) that confirmed a rise of the sintered density of 2017 Aluminium alloy matrix with the increase of the sintering combinations.

In addition, the pores became infrequent as the densification behavior progressed. But, residual porosity was evident throughout the SEM image. For instance, as shown in figure 6(a), the amount of porosity was higher in the microstructure sintered for (t1, T1) compared to the sintered for (t2, T2) as shown in figure 6(b).

Besides, the pores diameter decreases and it transformed from large size (figure 6(a)) into smaller size (figure 6(b)).

Concerning the effect of graphite content, analysis of the microstructure for the composite sintered at the highest sintering parameters combinations (figure 7) are conducted with different graphite content. Figure 7(a) shows the denser microstructure of 2017 Aluminium alloy matrix due to the use of higher sintering time and temperature (t2, T2). In addition, figure 7 displays also the microstructures of composite samples, sintered under the same conditions (t2, T2), after the addition of different content of Graphite (b) 5 wt% and (c) 10 wt%.

The clear regions correspond to 2017 Aluminium alloy matrix, while the dark region correspond either to the graphite particles, indicated by red circles or to the pores which are indicated by white arrows.

Furthermore, the microstructure of composites with 5 wt% of Graphite (figure 7(b)) reveals that there is a non-uniform distribution of Graphite particles with some clustering at some regions which results the formation of pores at the 2017 Aluminium alloy and Graphite interfaces.

The appearance of porosity is in agreement with the results acquired in previous studies [38–40]. This behavior will affect the denser character of composites which is confirmed by the decrease of the sintered density.
and the rise of porosity presented in figure 5. This might be due to low density of Graphite particles (2240 Kg m$^{-3}$) as compared to that of 2017 Aluminium alloy matrix (2790 Kg m$^{-3}$).

As graphite content was increased to 10 wt% of Graphite (figure 7(c)), the porosity will have a relevant number. This result is consistent with the larger drop of the sintered density (figure 5(a)) and the increase of the porosity (figure 5(b)) with 10 wt% of Graphite.

3.3. Coefficient of thermal expansion (CTE)

For the measurement of thermal expansion, referring to sections (3.1 and 3.2), we will choose the sintering couple ($t_2$, $T_2$) which offers the highest densities of the composite. The effect of graphite content on the CTE of composites was analyzed by determining the thermal displacement versus temperature variations via dilatometry measurements.

Figure 8 shows the thermal strain hysteresis behavior of 2017 Aluminium alloy matrix composites with different content of graphite during continuous cycle of heating and cooling between 42°C and 500°C. Thermal hysteresis strain was represented as the displacement difference between the heating and the cooling curves after thermal cycle (figure 8).

We note that the thermal response curve of the unreinforced 2017 Aluminium alloy matrix exhibits hysteresis behavior due to the thermal stress. Such a phenomenon occurs when this thermal stress is higher than the yield stress of 2017 Aluminium alloy matrix which can induce after thermal cycle a residual strain [37]. This thermal hysteresis behavior can be explained by the large internal stress released and the high defect density (dislocations, porosity) in the matrix. This is proven in the literature for both powder metallurgy [46] and stir casting [47] processes.

With the addition of graphite particles within 2017 Aluminium alloy matrix, it is noticed from figure 9 that the thermal hysteresis of the composite has lower amplitude than that of the 2017 Aluminium alloy matrix after thermal cycle.

It is clearly shown on table 3 that with the addition of graphite particles, there is a rise of hysteresis parameter $\Delta \varepsilon$ and a drop of $\varepsilon$ strain value. This is explained by the increased resistance of expansion of the Al 2017 matrix provided by the reinforcement with graphite particles.

Firstly, the 2017 Aluminium alloy has undergone tensile stress and during the heating process, this stress was relieved with temperature [48]. As in mean time, the compressive stress in the matrix began to build-up and accumulate with the temperature because of unmatched expansion between SiC particle and matrix. This compressive stress arising in the matrix could exceed the yield strength of the matrix which lessens with temperature, thus ensuring the plastic relaxation [46]. During cooling process, tensile stress in the matrix re-builds-up again which could enlarge the distance between 2017 Aluminium alloy atoms and consequently

Figure 8. Displacement in 2017 Aluminium alloy composites during heating and cooling cycles for different Graphite contents.
reduce its expansion ability [46]. The compressive stress on the reinforcement enhance the restriction effect on the matrix and a new residual stress will be generated.

By summarizing, introducing graphite induced residual strain in the 2017 Aluminium alloy and this residual strain will be lower with the content of graphite particles (table 3) compared with unreinforced 2017 Aluminium alloy. According to Shui [46], more residual stress in the composite induces a larger hysteresis after thermal cycle.

As shown in figure 8, the displacement versus temperature curves of 2017 Aluminium alloy matrix composites with different content of graphite particles displays similar tendency after a thermal cycle. As mentioned previously, the plastic relaxation process was important with the increase of graphite content, resulting to a higher amount of residual stress and a large hysteresis after thermal cycle.

According to equation (6), the obtained CTE of composites were plotted versus content of graphite and shown in figure 9. Introducing a high amount of graphite particles allows the decrease of the CTE of composites. This gradual decrease in CTE values of composites (figure 9) can be enlightened by the lower CTE of graphite particles than 2017 Aluminium alloy matrix. Similar results were reported for the CTEs of AlSi7Mg, Al 2024 [49, 50] and Copper [46] based composites.

4. Conclusion

In the current study, 2017 Aluminium alloy chips were successfully recycled into secondary 2017 Aluminium alloy via the direct conversion method with powder metallurgy method. Then, recycled powder was reinforced with graphite particles to produce composites.

Through the physical characterization of composites with different graphite contents, it was found that the addition of graphite particles generates a decrease of density due to the lower density of graphite compared to the 2017 Aluminium alloy. However, it was found that with the increase of both temperature and time during the sintering process, the sintered density of the composite increased with the presence of a little porosity confirmed by SEM analysis.

It is a key material property especially when a composite structure works in a tempera-

The variation of displacement of 2017 Aluminium alloy and its composites with 5 wt% and 10 wt% of graphite were determined at temperatures varying from 42 °C to 500 °C for a single thermal cycle. As a result, the dimensional change of samples exhibits an increase with temperature, but, the addition of graphite particles greatly reduces the CTE of composites.
Based on the obtained results, it can be stated that the developed 2017 Aluminium alloy composite with different content of graphite is a good proposal for thermal management, then its thermal expansion is enhanced compared to the base recycled 2017 Aluminium alloy.

These results pave the way to our future works. Until now, we have determined the thermal expansion behavior of composites after the addition of graphite particles. Also, the extension of this work is to investigate the thermal conductivity (TC) which will be a subject of our future research.

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