Influence of warm temperature (0.3-0.5T_m) uniaxial compression on the distribution of reinforcing particles in a 2124-Al/10%SiC MMC

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Abstract. Experimental work was carried out to study the influence of uniaxial compression tests, performed on a Gleeble 3500, on the distribution of reinforcing particles in SiC reinforced Al Metal Matrix Composites (MMCs). Al MMC powder (2124-Al with 10 vol. %SiC) was produced through blending in a high energy ball mill. Unreinforced 2124-Al and Al MMC powders were compacted at ambient temperature (i.e. cold compaction) and the green compacts were then sintered at 490°C for 1 hr. Warm temperature (280°C) uniaxial compression tests were performed on a Gleeble 3500 thermomechanical simulator at a strain rate of 5 s⁻¹, strain of 0.3 and a soaking time 20 minutes. Microstructures of the uniaxially compressed 2124-Al with 10% SiC MMC revealed that deformation had an influence on the distribution of reinforcing particles. Reinforcing particles were only distributed inside 2124-Al grains that had undergone deformation (evident through change in shape of grains). This finding supports statements made in literature regarding the positive influence of deformation on the distribution of reinforcing particles. Engineering stress-strain curves showed that reinforcing 2124-Al with 10 vol. % SiC had a positive effect on flow stress as the 10%SiC MMC had a flow stress of 153 MPa while the flow stress of the unreinforced 2124-Al was 95 MPa. An improvement in hardness was also observed where theMMC had a higher hardness than the unreinforced Al alloy; further indicating that reinforcing the Al alloy improved properties.

1. Introduction
Metal matrix composites (MMCs) are still seen as viable alternatives to conventional metals and alloys for applications which require high temperature performance as well as high stiffness and strength [1]. MMCs may be continuously reinforced with fibres, or discontinuously reinforced by dispersing particles or whiskers with higher hardness in the matrix material [2]. Discontinuously reinforced MMCs are favoured over continuously reinforced MMCs due to lower cost [3]. The focus of this work is particle reinforced MMCs. One of the determining factors for producing particle reinforced MMCs that are of good quality is achieving a uniform distribution of reinforcing particles in the matrix [4].

Literature states that the fabrication process selected significantly influences the distribution of reinforcing particles in MMCs [5]. Powder metallurgy (PM) processes have been said to surpass liquid
state processes in their ability to achieve a uniform distribution [6]. However, many studies have reported the formation of a necklace microstructure after the blending and compaction steps in PM processes[5][7][8]. In this necklace microstructure, the reinforcing particles cluster on the periphery of the matrix grains along the grain boundaries as illustrated in Figure 1 [5]. This is undesirable because an MMC with this necklace microstructure is susceptible to intergranular cracking which may result in failure [9]. As a result, PM processes consist of a secondary processing step where a deformation process (e.g. uniaxial compression, extrusion or drawing) is used to induce plastic flow in the MMC in an attempt to improve reinforcing particle distribution [10].

![Figure 1. Typical necklace microstructures observed in literature by a) Prasad et al. (2002), marker = 10µm, b) Liu et al. (2010), marker = 20µm and c) Chen et al. (2015), marker = 10µm.](image)

Deformation temperature is a critical factor. Hot deformation (i.e. temperatures higher than 350°C) is associated with inferior quality due to poor dimensional accuracy and defects [11][12]. The presence of reinforcing particles increases resistance to deformation which makes deforming MMCs at ambient temperatures cumbersome and impractical for industrial applications; especially where mass production is required since the service life of equipment and die sets is adversely affected and their frequent replacement increases production costs [12]. Most deformation processes are carried out at temperatures above 350°C (hot deformation). There is therefore a knowledge gap to be filled regarding deformation at intermediate or warm working temperatures.

This work aims to investigate the influence of warm temperature uniaxial compression on the distribution of reinforcing particles in powder metallurgically produced 2124-Al/10%SiC MMCs. A part of the work is to study microstructures (evolution from compaction to uniaxial compression) and assess if statements about the positive influence of deformation on distribution of reinforcing particles, made in literature, can be corroborated.

2. Experimental procedure
An Enerpac VLP® 100 tonne hydraulic press and 8 mm diameter die set made from high speed steel (HSS) were used to compact unreinforced 2124-Al alloy and blended 2124-Al with 10 vol. %SiC powders to form green compacts. Ambient temperature compaction was carried out because the limitation of this Enerpac press is that powders can only be compacted at ambient temperature. The green compacts were sintered at 490°C for 1 hr prior to uniaxial compression. To determine the warm working temperature range, a differential scanning calorimetric (DSC) study was done on the unreinforced 2124-Al and MMC. The DSC results showed that the materials started melting at ~560°C (solidus temperature). This meant that the warm working temperature range (30 and 60% of the matrix phase melting point) was between 168°C and 336°C. In a previous study, it was observed that at temperatures below 280°C the green compacts fractured; indicating that deformation was poor. Uniaxial compression tests were performed on a Gleeble 3500® thermomechanical simulator at 280°C, strain rate of 5s⁻¹, strain of 0.3 and soaking time of 20 minutes. To evaluate the influence of this uniaxial compression on the distribution of the reinforcing particles, the sample preparation technique was critical. Sample preparation involved only one grinding step with a 1000 grit paper, at a force of 20N for 2 minutes. Only one grinding step was used because SiC papers are used for grinding and it has been observed that SiC particles from the grinding paper tend to adhere to the MMC samples and may be mistaken for the SiC particles that were
added. The focus of this work is evaluating the influence of processing steps, particularly uniaxial compression, on the distribution of reinforcing particles. Therefore, the possibility of mistaking SiC particles from the grinding paper for those that were added had to be eliminated so as to acquire a good representation of the influence of the processing steps on reinforcing particle distribution. The grinding step was followed by polishing using 15 µm, 3 µm and 1 µm polycrystalline diamond paste using a force of 20 N for 5 minutes at each step. The samples were then polished with colloidal silica at 15 N for 15 minutes and water for 10 minutes. A Leica DM15000M® optical microscope and Jeol-6510® scanning electron microscope (SEM) were used to analyse the resultant microstructures. Microhardness tests with a 100g load were done on the compacts after uniaxial compression tests using a Future-Tech Vickers microhardness tester FM-700®. Fifteen indentations were made on each sample across the diameter of the compacts using a distance of 0.200 mm between indents. An average of the fifteen hardness values was calculated to determine hardness for each sample. Hardness tests were carried out according to ASTM standard E92 (ASTM E92-17, 2017).

3. Results and discussion
3.1. Blending & compaction
3.1.1. Unreinforced 2124-Al

Figure 2 shows that the unreinforced 2124-Al alloy powder particles were spherical, the powder compacted well and no defects (e.g. surface cracks) were observed on the green compacts. Microstructural analysis revealed that the microstructure of the 2124-Al green compacts was inconsistent from the surface to the mid-height (also referred to as the core). The darker phase in the microstructure from the core represents the pores and it can be seen that there is more porosity (as well as larger pore size) at the core of the green compacts. This inconsistent microstructure illustrates that there was a density variation in the green compacts due to pressure gradients that are caused by the friction between the powder and the die walls. It can also be observed that the Al alloy particles, under the compressive load, adhered to one another, bonded relatively well and showed good consolidation overall.

Figure 2. Images showing as-received unreinforced 2124-Al alloy powder (marker =100µm), 2124-Al green compact as well as resultant microstructures at the surface and core of green compact (marker = 250µm).

3.1.2. 2124-Al with 10%SiC

The microstructure showing results from the blending step in Figure 3 shows that there were some SiC particles which adhered to the Al alloy particle surfaces and a considerable amount of loose SiC particles situated around the Al alloy particles. These loose SiC particles indicate that the SiC powder did not blend fully with the Al alloy powder. Compaction of the blended powder was successful and green compacts produced were of good quality and comparable to those of the unreinforced Al alloy. Microstructures show that the typical necklace microstructure (Figure 1 compared to Figure 3) was
formed after cold compaction. When one analyses microstructures after blending and compaction one can infer that, in this context, this necklace microstructure formed as result of poor blending and type of compaction used. Figure 4 illustrates that as the load applied during compaction forces the Al alloy particles to move closer together, the loose SiC particles (together with those attached to Al alloy particle surfaces) were caught in between the Al alloy particles. As a result, as the Al alloy particles consolidated the SiC particles agglomerated in between them. The SiC particles were then stuck at the periphery of the Al alloy grains once they had consolidated because plastic flow was limited (due to cold compaction) and there was no mechanism for the SiC particles to move from the grain boundaries to the inside of the grains. This result highlights that, where process costs are not a major concern, hot compaction should be considered to increase plastic flow and possibly improve distribution of reinforcing particles in compacts. It was also observed that sintering had no influence on the distribution of reinforcing particles and this makes sense because conventional sintering, which does not involve the use of pressure, was used. The white phase inside the Al alloy grains represents the eutectic.

Figure 3. Images showing blended 2124-Al with 10%SiC powder (marker=50µm), 10%SiC green compact as well as resultant microstructures at the surface and core of green compact (marker=50µm).

Figure 4. Image showing mechanism by which SiC particles agglomerated on Al alloy grain boundaries during cold compaction taken from Prasad et al. (2002).

3.2. Uniaxial compression
Figure 5 illustrates that reinforcing 2124-Al alloy with 10%SiC improved flow stress significantly because the 2124-Al with 10%SiC MMC had a higher flow stress of 153 MPa when compared to the unreinforced Al alloy whose flow stress was 95 MPa. It can also be observed that the MMC was able to deform at the maximum flow stress of 153 MPa up to the maximum strain of 0.3. This result is good considering that MMCs have been reported to be more difficult to deform than their unreinforced counterparts [14]. Moreover, this good deformation was achieved within the warm working temperature range at a temperature of 280°C.
Figure 5. Stress-strain curves showing compressive behaviour of the unreinforced 2124-Al alloy and 2124-Al with 10% SiC MMC at 280°C, 5 s⁻¹, strain of 0.3 as well as sintering at 490°C for 1 hr and 20 minutes soaking time prior to uniaxial compression.

Figure 6 shows microstructures of the 2124-Al with 10% SiC MMC after uniaxial compression. The microstructures clearly show that the distribution of reinforcing particles was only influenced and slightly improved in areas where deformation had occurred. Deformation is identified by the Al alloy grains’ change in shape. It can be observed that where the 2124-Al alloy grains deformed, as a result of uniaxial compression, some of the SiC particles migrated from the grain boundaries to the inside of the Al alloy grains and these areas are shown with white circles in Figure 6a. This result, without a doubt, corroborates statements made in literature regarding the positive influence of deformation on the distribution of reinforcing particles [5]. This finding also illustrates that there is potential to improve the distribution of reinforcing using a deformation process such as uniaxial compression; however in this case it seems that more deformation was required. It can be inferred that plastic deformation of the Al alloy grains is the mechanism by which distribution is improved by deformation processes since, from the microstructures shown in Figure 6, it is clear that in cases where the Al alloy grains did not plastically deform the SiC particles remained on the grain boundaries. It can further be deduced that the necklace microstructure that is often acquired after compaction in powder metallurgical processes can be improved with the aid of a deformation process that induces sufficient deformation to the MMC. In Figure 7 the SiC particles can be clearly seen inside the deformed Al alloy grains. The white square in Figure 7a shows the spheroidised eutectic (as a result of sintering) which should not be mistaken for smaller SiC particles [15].

Figure 6. SEM BEI images showing microstructure of the uniaxially compressed 2124-Al with 10% SiC at a) 300X magnification (marker = 50µm), b) 500X magnification (marker = 50µm) and c) 1000X magnification (10µm).
3.3. Hardness

Hardness results after uniaxial compression (Figure 8) show that the MMC had higher hardness when compared to the unreinforced Al alloy. The higher hardness in the MMC was attributed to the presence of reinforcing particles. Reinforcing particles are believed to improve hardness of materials by improving load bearing capacity through the load transfer effect. The load transfer effect explains why MMCs (and composite materials in general) are able to bear larger loads than unreinforced materials. According to the load transfer effect, any load that is applied to composite materials is shared between the matrix material and the reinforcement. The reinforcement is stiffer and stronger and is able to bear larger loads than the matrix material. This therefore explains why composites tend to have higher hardness values when compared to the unreinforced material[14].

It can also be observed that there is a variation in hardness from the surface to the core in both the materials. The hardness of the unreinforced 2124-Al was found to be 51.60 HV\textsubscript{0.1} and 59.16 HV\textsubscript{0.1} at the surface and core respectively. Hardness values of the MMC at the surface and core were 85.73 HV\textsubscript{0.1} and 97.96 HV\textsubscript{0.1} respectively. A higher hardness was observed at the core of the samples and it was thought that this could be due to better densification at the core of the samples during uniaxial compression. There seems to be a relationship between density and hardness since density measurements showed that the 10\%SiC MMC had a higher relative density (99.431\%) than the unreinforced 2124-Al (95.072\%).

Hardness was measured at the surface and core of the samples to evaluate if hardness was consistent from the surface to the core. This inconsistency in hardness implies that there are inconsistencies in densification from the surface to the core, which was observed during the compaction stage and is a well-known phenomenon in compaction. This result is significant because it highlights how complex the deformation process is during uniaxial compression and also shows that deformation can be inconsistent across the length of deforming compacts. It can then be inferred that this variation in hardness implies that resistance to applied load is not uniform. This means that for optimal performance of these materials, this variation in hardness should be taken into account. Furthermore, this result illustrates that surface hardness measurements may not necessarily give a true reflection of the hardness of a sample or component.
Figure 8. Hardness of surface and core of uniaxially compressed unreinforced 2124-Al and 2124-Al with 10%SiC MMC samples.

4. Conclusions
The influence of warm temperature (0.3-0.5Tₘ) uniaxial compression on the distribution of reinforcing particles in a 2124-Al/10%SiC MMC was studied and the following conclusions were drawn:

1. Deformation, as a result of uniaxial compression, had a positive influence on the distribution of reinforcing particles since an improvement in distribution of reinforcing particles was only observed in grains that had undergone deformation. This finding corroborated statements made in literature regarding the positive influence of deformation on distribution of reinforcing particles.

2. Reinforcing the 2124-Al alloy with 10 vol. % SiC particles improved flow stress since the 10%SiC MMC had a flow stress of 153 MPa while flow stress of the unreinforced 2124-Al was 95 MPa.

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