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High temperature superplasticity and its deformation mechanism of $AA6063/SiC_p$



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ABSTRACT

Superplastic forming is primarily used for thin-walled and complex shape structures. This process is much successful in industries mainly for the fabrication of intricate components used for aerospace and automobile industries. In this paper, superplastic forming behavior and microstructural evolution in Al/5%SiC_p composites were studied at different temperatures through the hot tensile test. In a hot tensile test, a maximum elongation (i.e., 227%) was obtained at the 580 °C temperature condition. When the temperature was more than 580 °C, the elongation was reduced to less than 100%. Similarly, when the temperature is adjusted to a value lesser than 550 °C, the reduction in the elongation is seen which is less than 100%. Here, the microstructure study of the test samples is studied through the optical microscope. From the microstructure analysis, the grain refinement is taken place by dynamic re-crystallization and the intergranular deformation with grain boundary sliding is observed. It is understood that, the dynamic recrystallization and grain boundary sliding increases the percentage of elongation and on the other hand the ductility reduces due to the presence of excessive liquid phase at the grain boundaries.

1. Introduction

Discontinuously strengthened metal matrix composites are engaging for several structural applications due to their high specific strength and their modulus of physical property. Discontinuously reinforced aluminum (Al) metal matrix composites having superplastic forming behavior at high strain rates are most preferred in many industrial applications. In industries, superplastic forming has been done successfully in the fields of aerospace (say for example aircraft structures) and automobile industries [1,2]. For the past two decades superplastic forming playing a major role in manufacturing industries. Superplasticity could be a development exhibited by certain metals that expresses outstanding plasticity beneath specific conditions of forming [2–4]. Superplastic forming presents probably an engaging alternative to alternative forming techniques, and this is due to the low flow stress characteristic of superplastic deformation, and low tooling price. The tensile plasticity of Al composite strengthened with silicon carbide particles (SiC_p) has shown the concern of elongation up to 300%. Blow kindling is often accustomed form metal sheets beneath superplastic conditions. Superplastic behavior of a composite is primarily assessed by mistreatment either uniaxial or line testing. Research has been conducted to check and appraise the superplastic behavior of Al/SiC_p at high strain rate [1,4]. Mechanical properties like enduringness and hardness tend to induce at elevated temperature [5]. Studies have shown enhancements in material properties with improved creep

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resistance in certain elevated temperatures [5]. When a weak metal, like Al, is concerned, strength is an important parameter to be concentrated, and the addition of SiCp achieves the required strengthening. The main mechanism of superplastic forming is *Grain Boundary Sliding* (GBS). Smaller grain size is simpler to rotate and slide over each other, to accommodate giant strains before failure. However, superplasticity in coarse-grained metal alloys has been rumored. Al, with 36 µm grain size, conjointly was reported. Superplasticity is often achieved even with a grain size of 10 µm. By combining the consequences of each (the grain size and temperature), superplasticity is often achieved at little grain size and low temperature or large grain size. These microstructural changes are marginally affecting the mechanical properties of the material [6–8].

In a study, Aluminum–Lithium 2195 alloy having low density with better mechanical properties was used for creating a hemispherical dome by using the superplastic forming. In their hemispherical design they were able to maintain the superplastic conditions at 460 °C for the 350 mm diameter dome during the forming process [20]. Xiao-guo Wang et al., reviewed the superplastic formation behavior of Al Alloy, where the they summarised the fundamentals related to superplastic deformation of Al alloys (Al-Mg alloys, Al-Li alloys, Al-Zn-Mg alloys, and other Al matrix composites) [21]. In another study, the hot tensile tests were carried out on the superplastic tensile specimen prepared out of 7B04 Al-alloy. It is revealed that at around $470 \sim 530$ °C, the initial strain rates are observed, and they were in the range of 3×10^{-4} s⁻¹ to 1×10^{-2} s⁻¹ [22]. Another study, on the 7B04 Al alloy under similar condition as mentioned in Ref. [22], revelled that with an increase in temperature there is a decrease of strain rate. This intern decreases the flow stress of 7B04 Al alloy there by allowing the % of elongation to increase [23].

In this paper, aluminum alloy 6063 is considered. The study was aimed to conduct experimental analysis to understand the superplastic behavior and microstructural analysis of Al/5%SiCp composites under high temperatures. The manuscript is structured as follows: section 2 provides the experimental details about tensile sample preparation, hot tensile test, and microstructural analysis. In section 3 results were discussed and finally the concluded in section 4.

2. Experimental method

2.1. Tensile sample preparation

Aluminum alloy 6063 is cut and weighed to obtain the correct weight as per the stoichiometric calculations. The furnace is heated to a temperature of 800 °C and is constantly maintained at that temperature throughout the process. The silicon carbide (SiC) particles are preheated to a temperature of 1000 °C before it is mixed with the molten Al alloy. The heat treatment on SiC particles was done in order to form a layer of silicon dioxide (SiO $_2$) on the SiC, which improves the incorporation of the SiC $_p$ into the molten melt [9]. The material is then stirred by maintaining the string speed as 300 rpm for around thirty minutes. The stirred molten metal is then slowly poured into the die which is preheated to a temperature of 973 °C. The die is allowed to cool in the air for two hours.

For enhancing the superplastic formability, the material is allowed to undergo several advanced forming techniques like Friction Stir Processing (FSP), Equal-Channel Angular Pressing (ECAP), High-Pressure Torsion (HPT), and Accumulative Roll Bonding (ARB) [21]. By these severe plastic deformation techniques fine grains, high superplasticity, and low flow stresses are obtained for the materials. In this work, the casted specimens are thermo-mechanically treated to refine the grains and remove porosity. During the thermomechanical treatment, the specimen thickness is reduced from 5 mm to 2 mm. Fig. 1 shows the tensile specimen (sub size) for hot tensile test [10].

2.2. High temperature tensile testing of the specimen

High temperature tensile tests were done, using the specimens which were made from the composite sheets. Tensile test was

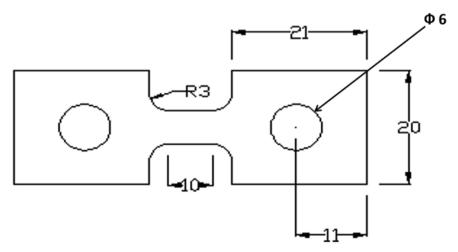


Fig. 1. Sub size of the tensile test specimen.

conducted using Instron Universal Testing Machine with the attachment for heating. The tubular furnace was used to heat the specimen. The tensile tests were conducted at different temperatures of $520\,^{\circ}$ C, $540\,^{\circ}$ C, $560\,^{\circ}$ C, $580\,^{\circ}$ C, and $600\,^{\circ}$ C and the constant strain rate of $0.2s^{-1}$. Once the test is done, and after the specimens were broken down, they were quenched in water to retain the microstructure during fracture. The fractured specimens were prepared with the hot setting process and polished with different grade sheets. These specimens were mirror polished with alumina powder by using a rotating velvet cloth disk, and the Keller's solution was used as an etchant.

Fig. 2. (a) shows the *Instron Universal Testing Machine* attached with a tubular furnace with a temperature controller. In order to understand the stress-strain diagrams, the *Universal Testing Machine* was connected with the computer where the data processing is done. The specimens were fixed between the two arms as shown in Fig. 2. (b) which will be connected to the upper and lower part of the machine. After the specimen was fixed between the two arms, then the furnace should be closed. The required temperature was set in the controller. When the temperature reached the specified value, it was maintained another 10 min for the equilibrium condition of the specimen. From the hot tensile test, the actual stress-strain curve was drawn. The fractured specimens are shown in Fig. 3. (a) were carefully assembled and the gauge lengths at fracture were measured, and the percentage of elongation was measured for various temperatures using Eq. (1).

% of elongation =
$$\left(\frac{G_{laf} - G_{lbf}}{G_{lbf}}\right) \times 100$$
 (1)

Where, the G_{laf} is the gauge length after the fracture, and G_{lbf} is the gauge length before the fracture.

2.3. Microstructure analysis

The structure analysis is carried out by studying the microstructure of the test samples which were taken from the grip region to the broken end. The microstructures were studied under-etched condition using an optical microscope. The image or the micrograph was captured for analysis using Biovis Material Plus software. The positions of the hot tensile samples where the micrographs were taken are shown in Fig. 3. (b).

3. Results and discussion

The uniaxial superplastic forming tests were conducted through hot tensile test for different testing temperatures. In general, these uniaxial tensile tests were carried in characterizing the material behavior, and the procedure seems to be quite straightforward. However, the most uncomplicated testing procedure of tensile test will be become a complex one due to the heat involvement. In our study, hot tensile tests were carried out to study the behavior of Al6063/SiC_p composite material at different temperatures. After the test, the fractured specimens were carefully assembled at the gauge lengths and elongations were measured. The stress and strain curves for different temperature were also plotted. The flow and fracture properties derived from the tensile test was strongly dependent on the temperature at which the test was conducted. In general, when the strength decreases, and ductility increases as the test temperature is increased. However, there is a possibility for structural changes especially at a specific range of temperatures. Generally, these structural changes are referred to as microstructural changes that include the precipitation, re-crystallization, strain aging which will eventually alter the metal behavior. The effect of deformation temperature on the actual stress versus strain curves for Al6063/SiC_p composites is shown in Fig. 4.

The increase in temperature decreases the flow stress at all the temperatures, and strain hardening is observed to occur almost up to the point of failure and relatively little strain softening is observed [11]. According to the power law of equation, the flow stress of a material at high temperatures should increase with an increase in grain size. It is considered that the strain hardening behavior of the composite is related to its concurrent matrix grain growth during deformation. The grain growth has been discussed using the fractography study. After the thermo-mechanical treatment, an equal amount of grain was formed. The average grain size was 25 μ m. Hot tensile tests were carried out at 560 °C, 580 °C, and 600 °C at the strain rate of 0.02s⁻¹. Maximum elongation of 227% was obtained at 580 °C for the specimen D. The further increase in temperature reduces the ductility because of the presence of excessive liquid phase at grain boundaries. Only 98% of elongation was obtained at the temperature of 600 °C. Maximum elongation was obtained at 580 °C. Table 1 shows the percentage of elongation at different temperatures, and the deformed specimens tested at various temperature of the hot tensile test are shown in Fig. 3. (a).

The microstructures were taken from the maximum elongated specimen at six positions typically from the grip area to the fractured tip area. The microstructure of the developed composite (a deformed specimen when tested in hot tensile test at the temperature of 580 °C at the strain rate of $0.2s^{-1}$) is shown in Fig. 5. The figures were modified into color format through Gwyddion software¹. Here, the blue color represents the aluminum matrix and the red color shows the presence of SiC particles and the green color as the grain boundaries. The shown microstructures were taken and converted into a WLI image to show the matrix and the grain boundary at six different locations from the grip region to the wrecked region. Fig. 5. (a) shows the microstructure of the grip region and an equal amount of grains were found in the microstructure. During the heating conditions, the grain size was increased. In the gauge region, the grain size was smaller than the grip region. Many fine grains were observed at the gauge region and they can

¹ http://gwyddion.net/



Fig. 2. (a). Universal testing machine with furnace attachment; (b). The specimen fixed between the two arms (the upper and lower part).

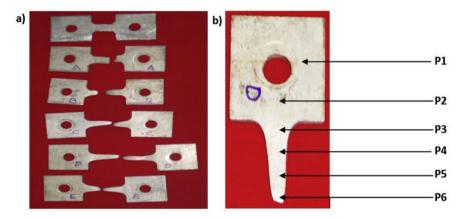


Fig. 3. (a). Original specimen along with deformed ones (tensile specimen samples A, B, C, D, and E) of Al6063/5%SiC composites at different temperatures; (b). Six positions at which the microstructures were taken.

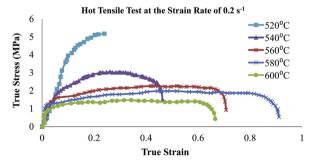


Fig. 4. Hot tensile test at various temperatures at a constant strain rate.

be taken as new grains that have been dynamically re-crystallized under the influence of stress at elevated temperature as revealed [11–15]. At the beginning of the deformation shown in Fig. 5. (a) the microstructures had transformed into a coarse-grained structure.

On the other side, Fig. 5. (b) shows some fine grains that started to form due to the stress and temperature. Fig. 5. (c) shows the grain boundary sliding mechanism. The superplastic forming has a mechanism like grain boundary sliding, dynamic grain growth, and grain rotation. The grain boundary sliding happens at low strain rates, and slipping occurs at high strain rate. The figure shows, the deformation proceeds by Grain Boundary Sliding (GBS). The average grain size was reduced to 36 µm, and the percentage of fine grains was increased to 25%. The microstructure shows that most of the fine grains are formed along the grain boundaries. It

Table 1% of elongation at different temperatures.

Tensile Specimen	Temperature (°C)	Elongation (%)
A	520 °C	24
В	540 °C	78
C	560 °C	153
D	580 °C	227
E	600 °C	98

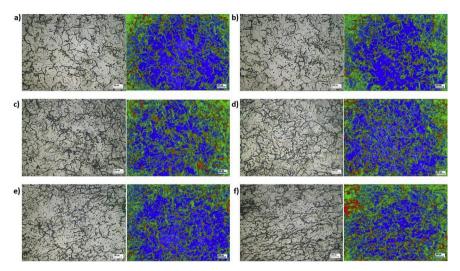


Fig. 5. Microstructure: (a). at position P1; (b). at position P2; (c). at position P3; (d). at position P4; (e). at position P5; (f). at position P6.

appeared that the stress assisted a dynamic grain growth which postulated that this process involved the migration of high-angle boundaries and the elimination of a large number of dislocations. Furthermore, it confirmed that the material exhibited two domains of dynamic re-crystallization [16–21]. At a temperature of 580 °C and the strain rate of $0.2s^{-1}$, re-crystallization was nucleated, and a fine grain size was produced. It was also found that many precipitates exist at the grain boundaries.

Fig. 5. (d) shows the middle portion microstructure of the specimen. Superplastic forming deformation increases from point C to Point D. 50% percentage of subgrains were formed. Dynamic recrystallization increases subgrain formation. The elevated temperature and stress lead to subgrain formation. The increase in the subgrain formation increases the superplastic formation. Large grains are not suitable for grain boundary sliding; small grains will improve the grain boundary sliding. Whereas, Fig. 5. (e) shows that subgrains that were oriented along the tension direction. During tension load, the stress was transferred from one-grain boundary to another. The grain boundary orientation makes the bands of Cooperative Grain Boundary Sliding (CGBS). The appearance of CGBS leads to the transition into plastic deformation. As soon as the stage of stable flow was reached, bands, start to appear as shown in Fig. 5. (f), and lead to the deformation. Generally, the more fine or elegant grains size increases the superplastic forming. As discussed earlier, the average grain size is relatively large at the beginning of the deformation. However, the larger grain size is not suitable for superplastic deformation, and on the other side, the dynamic re-crystallization process reduces the average grain size. The effect of second phase particles increases the dynamic re-crystallization [17]. The maximum volume of subgrains was formed at the temperature of 580 °C. Due to fine grains formation, an elongation as high as 227% was obtained.

4. Conclusions

The superplastic forming mechanisms were studied through the microstructure analysis of the hot tensile test specimens. The following conclusions were made:

- At lower temperature (the temperature considered in this study), the observed percentage of elongation was too minimum (< 100%), it was due to the absence of a liquid phase.
- The maximum elongation of 227% was obtained at 580 °C, the maximum volume of subgrains was formed at this temperature due to fine grain formation
- Further increase in temperature reduced the ductility because of the presence of excessive liquid phase at the grain boundary.
- The dynamic re-crystallization process reduces the average grain size, and on the other side, the grain boundary sliding increases the percentage of elongation.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.csite.2019.100479.

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