Research Article

Fabrication of Hybrid Surface Composite through Friction Stir Processing and Its Impression Creep Behaviour

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Al-Ni in situ surface composites were fabricated by friction stir processing method. Friction stir processing produced a composite with nickel and NiAl_3 as reinforcement particles in aluminium matrix. The particles were fine and were in the submicrometer size range. The separation distance between the particles was very small. Impression creep experiments were conducted on the samples both at friction stir zone and base material zone at various temperatures. Steady state creep rates were estimated, and activation energy for creep was calculated. It is observed that the friction stir zone offered a higher creep resistance compared to the base metal zone. Higher creep resistance is attributed to the dissolution of nickel atoms into aluminium matrix and the presence of fine nickel particles and NiAl_3 precipitates. The measured activation energy indicated that the associated creep mechanism is the dislocation creep in the temperature range of 30–150°C, both in friction stir zone and base metal zone. At higher temperatures (150–180°C) the diffusion creep mechanism is suggested.

1. Introduction

Aluminum and its alloys have versatile properties which make them suitable for use in a variety of applications [1]. In many of the applications, the surface needs to have better mechanical properties like improved strength and better hardness. Having a metal matrix composite at the surface is one of the options to have better resistance to wear, improved hardness, and even to an extent high-temperature stability at the surface [2]. Friction stir processing (FSP) is a new metal working method for producing surface composite. It is based on the concept of friction stir welding (FSW) [3]. During friction stir processing, the stirred material undergoes severe plastic deformation. The material flow associated with stirring and severe plastic deformation can be used for bulk alloy modification by the mixing of second elements, mixing followed by the precipitation of second phases, distribution of fine particles of second element, increased density of defects, and so forth. As a result, the stirred zone becomes a metal matrix composite with an improved hardness and wear resistance [4].

Arora et al. [5] have reviewed composite fabrication using FSP route. Mishra et al. [6] have used this route to make a

surface composite on AA 5083 alloy with 0.7 μm SiC particles. Shafiei-Zarghani et al. [7] and Mahmoud et al. [8] have incorporated Al_2O_3 and SiC particles to substrates like Al, Cu, and Fe alloys and observed an improvement in the abrasion and wear resistance of the surface composite. Ma and Mishra [9] have investigated the stability of the SiC reinforced aluminium matrix composite. Dolatkhan et al. [10] have investigated the mechanical and microstructural properties of Al-SiC metal matrix composites fabricated by FSP. Lee et al. [11] have investigated the microstructure and mechanical properties of Al-Fe in situ nanocomposite produced by friction stir processing. Hsu et al. [12] have reported the processing of Al-Ti and Al-Cu composites and in both systems formation of intermetallics is reported. Yadav and Bauri [4, 13] have reported the fabrication of aluminum matrix composite with Ni dispersion using FSP route. They have reported a composite without a detectable range of aluminides. Ke et al. [14] have produced Al-Ni intermetallics by friction stir processing and reported in situ formation of Al_3Ni and a mixture of Al_3Ni and Al_3Ni_2 after heat treatment. It also contained some Ni particles. Particle size range was varying from submicrometers to a few tens of micrometers. Qian et al. [15] have synthesised Al- Al_3Ni in situ composites using FSP route

and tried to explain its formation using concept of effective change in Gibbs free energy. They have also reported that the composite has better hardness and tensile properties.

To investigate the creep behaviour of these composites, impression creep testing is a suitable method [16]. Impression creep test offers several advantages over the conventional creep test. It takes a shorter duration for the test and only a small quantity of the testing material is sufficient. The temperature and stress dependency of creep rate could be obtained with a minimum number of samples. More importantly, it is the most suitable test method to study the creep behaviour of parent and processed zones independently [16]. To the best of our understanding, there is no report on the creep behaviour of Al matrix composite produced by FSP route. This paper reports the investigation of creep behaviour of friction stir processed Al-Ni surface composites investigated using impression creep technique.

2. Materials and Methods

Commercial pure Al sheet 5 mm thickness was used as the substrate material for friction stir processing. Base metal had coarse grains (average grain size $\sim 160 \mu\text{m}$) without any second phase particles (Figure 1(a)). Surface of the base metal was made rough to hold the powder during friction stirring. Approximately, 1 gram (per centimeter length of the sample) of electrolytic Ni powder was added on to the groove made on the substrate. Figure 1(b) shows the initial morphology of the nickel powder used. Ni powder was incorporated into the matrix using a friction tool made from tool steel. The following are the tool dimensions: shoulder diameter—20 mm, pin diameter—6 mm, and pin depth—3 mm. The following are the FSP parameters used: tool rotation speed—1200 rpm, tool travel speed—18 cm/min, and normal force—10 kN.

Using a precision sample cutting machine, the sample was cut perpendicular to the processing zone, and the cut surface was polished using standard metallographic techniques and etched to reveal the macrostructure. Figure 2 shows the macrostructure.

The macrostructural study reveals two distinct zones, namely, friction stirred zone and base metal zone. They are identified as FSZ (marked by A in Figure 2) and BMZ (marked by B in Figure 2), respectively. Using a scanning electron microscope (SEM), microstructural features were investigated both in FSZ and BMZ. Energy dispersive spectroscopy (EDS) attached to SEM was used for the investigation of chemical composition in FSZ. Phase identification in FSZ was done using X-ray diffraction (XRD) techniques.

Impression creep experiments were carried out both at FSZ and BMZ using a tungsten carbide indenter. A sketch of the tungsten carbide indenter used is shown in Figure 3. During experiments, the indenter was made to penetrate the sample (either at FSZ or BMZ). These locations are shown as A and B in Figure 2. A normal load of 5 kg was used during the impression test. On a 2 mm diameter contact area this gave a stress of about 15.6 MPa which is much less compared to yield strength of Al [17]. During impression experiments, the depth of the penetration of the indenter into the specimen was measured continuously using a linear

variable differential transducer (LVDT). Impression creep experiments were done for the duration of 180 minutes, and it is believed that this time is sufficient to attain steady state creep conditions. A plot of impression depth versus time (i.e., creep curve) was drawn. The creep experiments were conducted at various temperatures, namely, 30°C, 100°C, 150°C, and 180°C, for the purpose of estimating the activation energy and understanding the creep mechanism.

3. Results and Discussion

3.1. Microstructure and Phases in the Friction Stir Processed Zone. Figure 4 is a SEM micrograph from the FSP zone (A in Figure 2). The second phase particles are uniformly distributed without any clustering. Figure 5 shows a magnified micrograph from Figure 4. The SEM image in Figure 5 indicates that the particles show two types of contrast, namely, white contrast (B) and grey contrast (A). The chemical analysis using EDS indicates that the bright region (B) is nickel and the grey region (A) is NiAl_3 . It clearly indicates that the surface of the commercial aluminum substrate has changed to a hybrid composite using friction stir processing route. Pure nickel and nickel aluminide (NiAl_3) are the reinforcing phases in the surface of the aluminum. The nickel particles are added into the matrix by using FSP, and NiAl_3 has formed in situ during FSP. Also, EDS analysis in some locations in matrix Al indicates that some amount of Ni dissolved in Al. As much as 13% Ni is detected in some locations by SEM-EDS.

Particle size of a large number of particles as observed at a magnification of 2000x was measured using SigmaScan software (Jandel Scientific), and it is plotted in Figure 6. The particles are very fine and a major fraction of the particles has a size range which is less than $0.5 \mu\text{m}$. From Figure 5, it may be noted that the spacing between the particles is in the range of 2–3 μm . Figure 7 shows XRD plot of FSP zone. The data clearly shows presence of Ni in elemental form as well as in the intermetallic form (Al_3Ni).

Microstructural study along with EDX (Figure 5) and XRD analysis in the friction stirred zone (Figure 7) reveal the presence of undissolved Ni- and Al-Ni-based second phase particles. Al-Ni system has a number of intermetallics, namely, Al_3Ni , Al_3Ni_2 , Al_3Ni_5 , and AlNi_3 [18]. It is to be noted that the equilibrium solubility of Ni in Al does not exceed 0.04 at% [18]. The large extent of Ni dissolution in Al as observed during EDS analysis is attributed to the nonequilibrium nature of friction stir processing. Nonequilibrium phenomena during friction stir welding are reported by many investigators [19–21].

Yadav and Bauri [4, 13] have reported the fabrication of aluminum matrix composite with Ni dispersion using FSP route. They did not report the formation of any of the Al-Ni intermetallics during FSP. Formation of Al_3Ni during friction stirring is reported by Ke et al. [14] and Qian et al. [15]. Sieber et al. [22] have reported the reactive formation of NiAl_3 intermetallics at the interface of Al-Ni when sheets of Al-Ni are cold rolled and heat treated. They also observed a thin layer of Al_3Ni_2 at the interface of Al- Al_3Ni layer, after heat treatment.

FIGURE 1: Initial condition of the substrate and powder materials. (a) Microstructure of the Al substrate showing coarse grains; (b) initial morphology of the nickel powder used.

FIGURE 2: Macroview of the cross section of the friction stir processed sample (A), base metal zone (B).

FIGURE 3: Dimensions of the indenter used for impression creep experiments.

Arora et al. [5] have observed that large plastic strain in FSP can shear the metal powders. It can also break oxides on the surface of the particles, causing intimate contact between the matrix and the reinforcement particles, promoting reaction at the interface. The tendency for particles agglomeration is reduced as particles are covered by a layer of matrix material. High plastic strain imposed on the matrix material

FIGURE 4: Micrograph showing distribution of the particles.

FIGURE 5: A magnified micrograph from Figure 4 indicating two types of particles (A and B). The EDS analysis indicates that the particles are Ni (B) and NiAl_3 (A).

promotes particle size reduction and their distribution, and it also increases diffusion rate of elements, thus accelerating the reaction rate between the matrix and the particles [5, 15]. Extremely fine reinforcement particles act as pinning sites and help in controlling the grain structure during recrystallisation. FSP also provides a higher temperature to facilitate the formation of intermetallic phases in situ and accelerate the reaction [5, 21]. All these processes in tandem produce a surface with the dispersion of fine particles of nickel and nickel aluminide.

FIGURE 6: Particle size distribution of the reinforcing particles observed at 2000x magnification.

FIGURE 8: Penetration depth of the indenter as a function of time. FSZ, room temperature, and 5 kg load.

FIGURE 7: XRD analysis of friction stir processed region.

3.2. Impression Creep Behaviour. In the impression creep experiments, a calculated load was applied on the indenter positioned at the desired location in the sample (i.e., friction stir zone or base metal zone). The penetration depth was measured continuously as a function of time, and a creep curve of depth versus time is drawn. A curve for the conditions of room temperature and FSZ for the load of 5 kg is shown in Figure 8. Similar curves were generated both for FSZ and BMZ at various temperatures and were used in the analysis.

Using the values of penetration depth and indenter diameter, indentation creep strain was estimated for each curve, following the approach presented by Sastry [16]. Using these plots, steady state creep rates ($\dot{\epsilon}$) were estimated as follows:

$$\Delta\epsilon = \frac{\Delta l}{D}, \quad (1)$$

where $\Delta\epsilon$ is the incremental creep strain in the secondary stage of the creep curve profile and Δt is the corresponding incremental time, Δl is the incremental penetration depth, and D is the indenter diameter [16]. The values of steady state creep rate for the case of base metal and friction stirred regions at different temperatures are given in Table 1 (load:

TABLE 1: Steady state creep rates in friction stirred zone and base metal (load: 5 kg).

Temperature (°C)	Steady state creep rate (min^{-1})	
	Base metal region	Friction stirred region
30	3×10^{-5}	8.33×10^{-6}
100	30.4×10^{-5}	8.7×10^{-5}
150	200×10^{-5}	90×10^{-5}
180	235.7×10^{-5}	178.9×10^{-5}

5 kg). We see that the steady state creep rate value increases as temperature increases, and this is shown in Figure 9.

The following observations could be made from the data presented in Figure 9.

- (1) Creep rates are lower in the friction stirred region than the base metal region at almost all temperatures.
- (2) Creep rates are low at lower temperatures but they increased exponentially with temperature both for base metal and friction stirred regions.

Activation energy for creep (Q) is calculated by using the following equation:

$$Q = \frac{R \ln(\dot{\epsilon}_1/\dot{\epsilon}_2)}{1/T_2 - 1/T_1}, \quad (2)$$

where R is the gas constant and $\dot{\epsilon}_1$ and $\dot{\epsilon}_2$ are the steady state creep rates at temperature T_1 and T_2 , respectively. The values of the activation energy for the impression creep of the friction stirred region and base metal region were estimated, and they are in the range of 45 kJ/mole to 55 kJ/mole in the temperature range of 30–150°C. Comparing them with the activation energy of the movement of dislocations in Al alloys, it could be inferred that the underlying creep mechanism is the dislocation creep, both in the substrate and FSP zones. In the temperature range of 30–150°C, the thermal activation affects the lattice resistance to the glide of the dislocation. The temperature-dependent variation of the creep rate is related to the thermally assisted force required to overcome the obstacles lying in the plane of the dislocation glide. Thus, it is arguably inferred that the dislocation creep

FIGURE 9: Variations of steady state creep rate ($\times 10^{-5}$) as a function of temperature. $\blacklozenge$ Base metal region, $\blacksquare$ friction stir zone.

dominates the mechanism of creep in Al base metal and friction stirred zones. The lower value of activation energy is mainly attributed to the nonequilibrium nature of the material due to friction stir processing. Presence of a large amount of dislocations and very fine grain scales makes the dislocation glide easier, by providing a large number of closely spaced sinks (in the form of grain boundaries).

The activation energy determined is in the range of 140 kJ/mole to 170 kJ/mole in the temperature range of 150–210°C. It is very close to the activation energy for self-diffusion (143.4 kJ/mole) in aluminium [23, 24]. For this reason, rate controlling mechanism at elevated temperatures, in both the friction region, and base metal region has been ascribed to the diffusion creep. Diffusional creep is influencing creep behaviour [25, 26].

Higher creep resistance offered by the friction stirred region compared to base metal region is ascribed to the following reasons:

- (1) presence of nickel atoms into Al matrix by mechanical alloying,
- (2) precipitation of Al_3Ni precipitates,
- (3) dislocations created by friction stir processing.

Higher level of creep resistance is exhibited by friction stir processed zone compared to base metal zone even at an elevated temperature. It suggests that second phase particles play an important role in the creep mechanism over the temperature range investigated. They act as barriers for dislocation movement. The hindrance to the dislocation movement is related to the distribution of particles and interface between the particles and the matrix [20, 21]. Fine particles, small interparticle separation distance, and good particle matrix bonding are essential for strengthening. Yadav and Bauri [4] have reported that the interface in Al-Ni composite produced by FSP is good and it hinders the dislocation motion.

4. Conclusions

Hybrid surface composite was made on a commercial pure aluminium by incorporating nickel particles using the friction stir processing route. The composite region consisted

of fine nickel and nickel aluminide particles produced in situ during friction stir processing. The dispersed particles were fine and uniformly distributed in the processed region. Impression creep experiments were done using a tungsten carbide indenter of a 2 mm diameter at temperatures in the range of 30–180°C. Plots of indentation creep depth as a function of time were obtained, and these plots were used for estimating the steady state creep rate. It is observed that the steady state creep rate is lower in friction stirred zone compared to base metal zone at all temperatures. Activation energy for creep was estimated and it was observed that at the temperature range of 30–150°C, the dislocation creep is predominant, and at higher temperatures the diffusion creep is playing a major role.

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