Indentation Creep Behavior of Nugget Zone of Friction Stir Welded 2014 Aluminum Alloy

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Abstract. The present study is aimed at evaluating the creep behavior of the nugget zone of friction welded 2014 Aluminum alloy by indentation creep tests. Impression creep testing was carried out at different temperatures of 300\degree C, 350\degree C and 400 \degree C with stress 124.77 MPa, 187.16 MPa, 249.55 MPa using a 1.0 mm diameter WC indenter. Experiments were conducted till the curve enters the steady state creep region. Constitutive modeling of creep behavior was carried out considering the temperature, stress and steady state creep rate. Microstructural investigation of the crept specimen at 400\degree C temperature and 187.16 MPa load was carried out and found that the small precipitates accumulate along the grain boundaries at the favorable conditions of the creep temperature and stress, new precipitates evolve due to the ageing. The grains are broken and deformed due to the creep phenomena.

Keywords: Friction stir welding, Impression creep analysis, constitutive modeling, microstructural analysis

1. Introduction

Structural components for application in aviation industries and energy sector are subjected to elevated temperatures and non-steady loading conditions. A large number of components are welded and require stringent quality control before being put in to use. In welded components which subjected to extended periods of service at elevated temperatures, the main failure is by creep deformation. In recent years friction stir welding (FSW) process has revolutionized the quality of welding especially in difficult to weld materials such as high strength Aluminum alloys. The weld region of a friction stir welded component has mainly three zones: (i) nugget zone, (ii) thermo-mechanically affected zone and (iii) heat affected zone The nature of the material at the FSWed zone is highly non-equilibrium due to the
heterogeneity in the microstructure and chemical composition. There is a variation in the grain structure in the NZ, TMAZ and HAZ with respect to the base material [1]. The grain size at the NZ is smaller as compared to the other zones. On the other hand the NZ in the FSWed sample possesses finer grains as a result of recrystallization due to high mechanical deformation occurred as the stirring action of the tool pin and the heat generated due to the friction between the pin and the material to be welded [2-3]. So the finer the grains, the chance of generation of the dislocations are more. At the time of creep, near the grain boundary in the NZ a numerous generation of dislocations occur as a result of ‘Frank Reed Mechanism’ which interact with the inter granular dislocations. So there is a process of generation and interaction of dislocations occur due to which a dislocation cross slip occurs which promotes plastic strain at low temperatures and low stress values [4]. The mechanical properties and deformation behavior of these three zones are different and the final failure of the material is decided by the properties of the weakest zone. The general procedure followed is to study the creep behavior of the welded component on a macroscopic level by carrying out experiments using tensile specimen. To the authors’ knowledge, report regarding the study of the creep behavior of different weld zones in a friction stir welded components is not available in the literature.

In recent years, impression creep (IC) testing has been developed in which the creep deformation behavior of small localized areas of a component can be studied. In this technique, a constant load is applied on a disk shaped sample using a cylindrical indenter at a constant temperature. The time dependent penetration of the indenter in to the test specimen is measured and the constitutive relationships for the creep deformation are developed. In case of hot hardness testing, the creep behavior of the material cannot be correlated to the hardness since steady state is not achieved due to the increase in the indentation area with increase in penetration depth. Hence a constant stress cannot be maintained with penetration of the indenter. The drawback can be overcome by impression creep testing method where a uniform cross section indenter is used under uniaxial compression [5]. The main advantages of IC are: (i) the small sample size requirement, (ii) creep behavior of different zones similar to that of weld zones can be determined, (iii) the test process takes very less time and (iv) the data can be used for modeling the creep deformation of emerging materials [6-7].

Figure 1 illustrates schematically the impression creep testing procedure. A flat ended cylindrical indenter of diameter ‘a’ is placed on the top of the creep test specimen and a pressure ‘P’ is applied as shown in figure. The entire set up is kept inside a chamber maintained at a constant temperature and environment during the entire period of the test. The time dependent depth of penetration of the indenter is measured using a linear variable differential transducer and recorded.
The present work is a part of the ongoing research on the deformation behavior of friction stir welded 2014 Aluminum alloy. Indentation creep testing of the nugget zone of the FSWed 2014 Aluminum alloy was carried out at different constant loads and temperatures. The steady state creep rate is estimated and constitutive equation for the creep deformation is developed.

2. Experimental Procedure

2.1 Materials

The material selected for the experiment was AA 2014 with chemical composition Cu: 3.9-5%, Fe: 0.7%, Mn: 0.4-1.2%, Mg: 0.2-0.8%, Cr: 0.1%, Si: 0.5-1.2%, Ti: 0.15%, Zn: 0.25% and balance Al. The chemical composition of alloy was ascertained by energy dispersive x-ray analysis (EDAX). The results of the EDAX analysis is shown in figure 2.

2.2. Experimental method

Friction stir welding was carried out with the process parameters: (a) tool rotational speed 815rev/min, (b) welding speed 22 mm/min and (c) square tool geometry with pin length 5.8 mm and pin diagonal
length 6mm. Subsequent to FSW, samples having dimensions 20 mm x 9 mm x 6 mm were cut from the weld region. The IC specimen was dry polished followed by cloth polishing to obtain a very smooth surface. A very light metallographic etching with Keller’s reagent (2mL HF (48%), 3 mL HCL (conc.), 5 mL HNO₃ (conc.), 190 mL H₂O) was carried out to reveal the nugget zone (NZ) of the FSWed region of the sample used for IC test. Typical photographs of the welded plate, test sample and the tungsten carbide indenter is as shown in figure 3 (a), (b) and (c) respectively.

Impression creep testing was carried out at three temperatures: 300ºC, 350 ºC and 400 ºC and three different stresses: 124.77 MPa, 187.16 MPa and 249.55 MPa. Total 9 experiments were carried out. The indenter displacement was measured using an LVDT having an accuracy of ±1μm. Strain ε is calculated to be the ratio h/D.

The minimum creep rate $\dot{\varepsilon}$ was determined as the minimum value of the $d\varepsilon/dt$ plot. The constitutive equation for the creep deformation can be expressed by the power law relationship:

$$\dot{\varepsilon} = A \sigma^n \exp \left( -Q / RT \right)$$

(1)

where n is the stress exponent, A is a constant, Q the activation energy for creep, R the universal gas constant and T the temperature in K. value of n is determined as the value of the slope of log ($\dot{\varepsilon}$) vs log $\sigma$ plot. The activation energy for creep is determined by the slope of ln($\dot{\varepsilon}$) vs 1000/T plot.

The sample after creep deformation at elevated temperature was sectioned longitudinally and passing through the indented region. The sectioned surface was metallographically polished and etched to observe under an optical microscope (make: Carl zeiss, Model: Axiotech). The metal flow below the indenter during the indentation creep testing stage was investigated.

Figure 3 Photographs of (a) welded plate, (b) impression creep test sample after creep testing and (c) tungsten carbide indenter.
3. Result and Discussion

Plots of creep strain vs. time and creep rate vs. time obtained from the impression creep tests carried out at 400°C and 187 MPa stress are shown in figures 4 (a) and (b), respectively. The figure indicates three stages of creep: (i) the primary creep showing increasing strain at a decreasing rate as a sudden load is applied at the beginning of the experiment, (ii) the secondary creep region characterized by the minimum creep rate and followed by (iii) the tertiary creep indicating an increasing creep rate with time. The decrease in creep rate with time in the primary creep stage is due to the high applied stress compared to the internal stress in the material. The steady state creep rate in the present investigation was the minimum creep rate obtained from the creep rate vs. time plot. The almost constant creep rate in the secondary creep stage is achieved when the applied stress and internal stress are balanced. Though the tertiary creep rate increases at a faster rate, a gradual decrease in the slope of the creep rate is observed after a penetration depth of around 0.4 mm. This is attributed to an increase in the frictional resistance due to increase in the contact area between the indenter and the sample [7-8].

![Figure 4. Plot of (a) strain vs. time, and (b) strain rate vs. time plot for the 2014 Al alloy at 400 °C and stress 187 MPa using 1 mm diameter indenter.](image)

The minimum creep rate at different combinations of stress and temperature is presented in table 1. The result shows that at constant stress the minimum creep rate decreases with decrease in temperature. The minimum creep rate also increases with increase in stress. The stress exponent for the AA2014 was calculated from the log (\(\dot{\varepsilon}\)) Vs log (\(\sigma\)) plot for different temperature conditions as shown in figure 5 (a). From the slope of the linear fits of the plot the average value of the stress exponent \(n\) is determined as 4.64. From the slope of the ln (\(\dot{\varepsilon}\)) vs 1000/T plot, the activation energy for creep \(Q\) is determined at a constant stress. From figure 5(b), the average value of the activation energy is found to be 114 kJ/mol. Since the activation energy for self-diffusion in pure Aluminum was reported as 142 kJ/mol [9], the activation energy obtained in the present investigation lies close to this value. Therefore one can conclude that the steady state creep in the present alloy is controlled by climb of edge dislocations.
Figure 5. Plot of (a) log (\(\dot{\varepsilon}\)) vs log \(\sigma\) and (b) ln(\(\dot{\varepsilon}\)) vs1000/T for the 2014 Al alloy.

Table 1. Steady state creep rate at different temperature and load for AA-2014.

| Stress (MPa) | Temperature (ºC) | Steady state creep rate(1/sec) |
|-------------|------------------|-------------------------------|
| 124.77      | 400              | 2.290 \times 10^{-3}         |
| 187.16      | 350              | 4.466 \times 10^{-4}         |
| 249.55      | 400              | 5.531 \times 10^{-2}         |

Optical micrographs of the samples before the creep test is shown in figure 6 (a). Figure 6(b) shows low magnification micrograph of the sample after creep testing at 400 ºC and 187 MPa, which is just below the indenter tip. The microstructure at the NZ of the FSWed specimen before the IC test shown in figure 6 (a) has almost equiaxed grain with uniform distribution. From the flow lines shown in figure 6(b), the deformation zone appears to be almost spherical in shape just below the indenter. This is due to the dead metal zone just below the indenter and extrusion of metal through regions near the lateral surface of the indenter. Figure 6(c) shows the same region at high magnification. Features typical of recrystallization are evident indicating dynamic recrystallization. Features also indicate precipitation of second phase particles at grain boundary regions. When 2014 Aluminum alloy is exposed to elevated temperatures, CuAl₂ particles will precipitate from the matrix [10].
Figure 6. Optical micrographs of 2014 Al alloy (a) before the creep test, (b) after creep test (just below the indenter) and (c) slightly higher magnification after impression creep test.

4. Conclusion

The indentation creep behavior of the nugget zone of friction stir welded 2014 Al alloy was investigated at 300 °C, 350 °C and 400 °C and stresses 124 MPa, 187 MPa, 249.55 MPa. The stress exponent $n$, the activation energy for creep $Q$ for the power law creep was evaluated. The microstructural study of the sample after creep test at different positions below the indenter was carried out and results showed the recrystallization at the grain boundary. The precipitation hardening and grain coarsening was also occurred.

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