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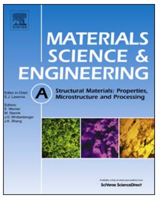

Grain refinement and superplastic behavior of carbon nanotube reinforced aluminum alloy composite processed by cold rolling

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ABSTRACT

The carbon nanotube reinforced aluminum matrix composite (CNT/6061Al) fabricated by flake powder metallurgy was cold rolled to reduce grain size, and was examined by high temperature tensile experiments within temperature from 325 to 450 °C and strain rate from 4.17E − 2 to 2.09 s^{-1} to explore the effect of grain refinement on the superplastic deformation behavior. The average grain size was reduced from 580 nm to 300 nm with better homogeneity after cold rolling, and elongation to failure at 400 °C and 4.17E − 1 s^{-1} was improved by 40% compared to that without cold rolling. The temperature of 375 °C and intermediate strain rate turned out to be appropriate deformation conditions for the refined microstructure, in which both large elongation to failure and uniform elongation was obtained. Stress exponent was changed to 2 and activation energy was determined to be 76.7 kJ/mol, indicating grain boundary sliding was the main deformation mechanism.

1. Introduction

Superplasticity is the ability of materials to show high tensile elongations prior to failure [1]. Currently, there is an interest in developing superplasticity in aluminum matrix composites in order to promote its superplastic forming capacity. Studies on a variety of aluminum matrix composites revealed that large elongation and high strain rate superplasticity (HSRS) were available at the temperature close or slightly above the incipient melting point [2–12]. Carbon nanotubes reinforced aluminum matrix composites are drawing increasing research interest from industries due to their high Young's modulus, high strength and low density. Our recent research on 1.5 wt% CNT/6061Al found out that although the largest elongation was obtained at 580 °C [13], the high temperature superplastic deformation should be avoided for CNT/Al system, because at such high temperature serious reaction between carbon nanotubes and the aluminum matrix would occur and lead to the formation of brittle intermetallic compound (Al_4C_3) [13]. In contrast, this material showed relatively better mechanical properties when the temperature was around 400 °C, attributed to the grain boundary sliding mechanism.

However, for the 1.5 wt% CNT/6061Al, the elongation at 400 °C with optimal initial strain rate was 89%, which was not as high as well-behaved superplasticity requires. A major task is therefore to achieve better superplastic elongation by adjusting the microstructures that affect the grain boundary sliding mechanism. Grain size is one of the most important microstructural parameters in predicting superplasticity. The decrease in grain size is expected not only to increase ductility, but also to increase the optimal strain rate and decrease optimal temperature for superplastic flow [10]. During high temperature deformation, a reduced and stable grain size has been reported to facilitate grain boundary sliding (GBS) and increase the ductility for superplastic materials [14,15]. Many severe plastic deformation methods [16], including equal-channel angular pressing (ECAP), high pressure torsion (HPT) and thermomechanical processing (TMP), etc. have been developed to introduce a high density of dislocation and eventually smaller grain sizes [1,17].

In the present experiments, cold rolling technique was used to reduce grain size of CNT/6061Al plate. The mechanical behavior and microstructure were then examined to explore the effect of grain refinement on the superplastic deformation. The improvement of the superplastic behavior and discussion about the mechanism are expected to facilitate the development of good superplasticity in carbon nanotube reinforced aluminum matrix composites.

2. Experiments

The CNT/6061Al composite was fabricated by a newly developed Flake Powder Metallurgy route via shift-speed ball milling [18]. The

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spherical 6061Al powder (about 30 μm, 99.8% in purity), 1.5 wt% multiwall CNTs (~ 20 nm in diameter, ~ 1 to 2 μm in length) and 1 wt % stearic acids were mixed and ball milled by a integration of low energy ball milling (135 rpm for 8 h) and high energy ball milling (270 rpm for 1 h). After degassing at 400 °C, the composite powder was consolidated by sintering at 550 °C for 2 h and hot extruded into a plate with the cross section of 60 × 12 mm at 480 °C. Then to further consolidate the composite, the plate was hot rolled into thinner plate with the thickness of 4 mm at 400 °C (denoted as hot-rolled specimen, to distinguish from the following cold-rolled ones). More details about the composite preparation can be found in previous paper [18]. Characteristic peaks for both CNTs and Al_4C_3 were detected by Raman spectrum in the hot-rolled composite, indicating that partial CNTs had reacted with Al matrix and formed intermetallic compound Al_4C_3. CNTs with length about a few hundred nanometers were found dispersed and laid at the grain boundaries by TEM observation [13].

To further reduce the grain size of CNT/6061Al, the hot-rolled plate with thickness of 4 mm was first rolled at 200 °C to 3 mm and then cold rolled at room temperature to 2.7 mm (denoted as cold-rolled specimen).

Tensile specimens were machined with a cross section of 4 × 2 mm and a gauge length of 4 mm in a direction parallel to the extrusion and rolling direction. Tensile testing was conducted by the computer-controlled electronic testing machine with three temperature sensors. Constant crosshead speeds were set for each test. All the specimens were heated for 30 min and held at the temperature for 5 min before the tensile testing. A range of strain rate from 10^0 to 10^{-2} s^{-1} and temperature of 325 °C to 450 °C were experimented. Scanning electron microscopy (SEM, FEI Scios) was conducted to examine the fracture surface under a voltage of 5 kV. Transmission electron microscopy (TEM, JEM 2100F) was used to character the grain size at a voltage of 200 kV.

3. Results and discussion

3.1. Microstructures

Fig. 1(a) and (b) show the typical TEM images for the specimens before cold rolling (hot-rolled) and after cold rolling (cold-rolled). Grain size was estimated by averaging the length and width of at least 50 grains in TEM images. It is found that the average grain size was reduced from ~ 580 nm to ~ 300 nm after cold rolling. Figs. 1(c) and 1(d) show the grain size distribution of these two specimens. We can see that not only the grains were refined but also the microstructure was more homogeneous after cold rolling. The hot-rolled specimen has a wide grain size distribution from 300 to 900 nm, while the cold-rolled specimen has a narrower grain size distribution from 100 to 500 nm. The superplastic deformation behavior of hot-rolled specimen has been studied in our previous paper [13], so in this paper we will focus on the cold-rolled ones. The result of hot-rolled specimen will be used for a comparison when we discuss the effect of grain refinement.

3.2. High temperature mechanical properties

The change in grain size is usually accompanied by other modification including misorientation degree of grain boundary, distribution of reinforcement and density and distribution of dislocations [19]. An overall evaluation of all the inner processes can be made by observing stress-strain curves. Mishia et al. [20] have suggested four categories of stress-strain curves: a classical well-behaved type, a continuous softening type, a continuous hardening type as well as a complex type. The well-behaved type with a plateau of stress means a more uniform elongation [21]. Grain coarsening, increase of grain boundary angle and dislocation entanglement are discussed to be the main reason for hardening. The formation and coalescent of cavity are supposed to be the main reasons for strain softening.

Based on the theory about categories of stress-strain curves, the true stress against true strain for cold-rolled specimens at different

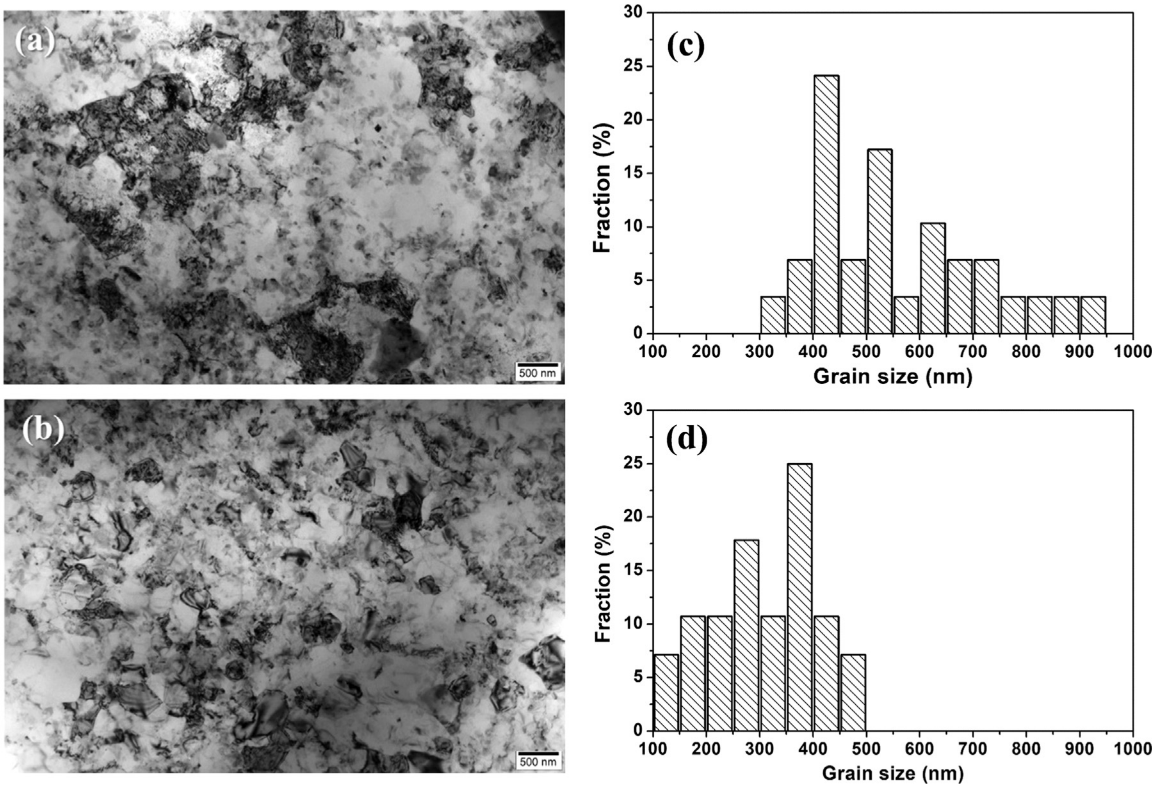

Fig. 1. TEM image for (a) hot-rolled and (b) cold-rolled CNT/6061Al; The distribution of grain size in (c) hot-rolled and (d) cold-rolled CNT/6061Al.