Materials Science and Engineering A 532 (2012) 567–572

Contents lists available at SciVerse ScienceDirect

Materials Science and Engineering A

journal homepage: www.elsevier.com/locate/msea

Analysis of creep fracture in Al–Al_4C_3 composite after ECAP

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ARTICLE INFO

Article history:
Received 2 May 2011
Received in revised form 24 October 2011
Accepted 25 October 2011
Available online 11 November 2011

Keywords:
Aluminum alloys
Equal channel angular processing
Mechanical alloying
Fracture

ABSTRACT

Creep fracture of a composite based on an aluminum matrix reinforced by 4 vol.% Al_4C_3 was studied at temperatures of 623 and 723 K by small punch testing with a constant force. The composite was tested in state after equal channel angular pressing (ECAP) by two passes of route C as a final operation. It was found that the time to fracture was inversely proportional to the minimum deflection rate in a similar manner as the corresponding quantities in conventional creep tests. The fractured surfaces of the studied materials had largely intercrystalline character.

1. Introduction

The mechanical properties of aluminum-based metal matrix composites in combination with their relatively low density have made them attractive for numerous applications [1]. It is clear that the method chosen for processing the composite has a strong impact on the final quality of the material. The present scientific research is intensively focused on the formation of nanoscale structures (with grain diameters $\leq$ 100 nm) in polycrystalline metallic materials, attained by severe plastic deformation (SPD). Materials produced by SPD are characterized by interesting values of strength, elongation, fatigue properties and superplasticity. The resulting properties are dependent on the nanoscaled structure, its distribution in the material, texture and other microstructural properties. The method of equal channel angular pressing (ECAP) [2–5] involves pressing the material through a die containing two channels of equal cross-section. Shearing strain depends on the geometry of the ECAP instrument, which is defined by the angle between the two separate parts of the channel, the angle on the outer corner of the die, the pressing speed, temperature, etc. The technique was applied successfully to pure aluminum [6–8] and aluminum alloys [9]. Little information is currently available on the application of ECAP to aluminum-based composites [10,11].

As a rule, only laboratory-scale samples of ECAP products are available for subsequent testing. Moreover, since the ECAP process offers a large variety of parameters, the conventional testing of products is very material-consuming. From this point of view, miniaturized methods [12] may be promising for the testing of ECAP products. Especially important is the Impression Creep test [13–15] with cylindrical indenter that gives a steady state penetration velocity at constant load. In usual conditions, fracture is not achieved in this type of test. Therefore, for the investigation of fracture mechanisms, the small punch test has to be preferred [16]. The method uses small disc specimens up to 10 mm in diameter with thicknesses up to 0.5 mm. The specimen is placed on a ring, and a ball (or a punch with hemispherical tip) is forced into the centre of the specimen. In the present contribution, the SP testing is applied to an aluminum-based composite prepared by the ECAP procedure. Effects of external parameters (temperature and loading force) as well as of time to fracture on the characteristics of fracture micromechanisms after "small punch" exposures are investigated with the aim to relate the results of the fractographical analysis with the shape of the small punch curves.

2. Experimental

Experimental material was prepared by the powder metallurgy route by the method of mechanical alloying. Aluminum powder with a particle size of <50 μm was dry milled in an attritor for 90 min with the addition of graphite KS 2.5 in an amount corresponding to 4 vol.% of Al_4C_3 in the resulting product. The mixture was then cold pressed using a stress of 600 MPa into compacts of cylindrical shape. Subsequent heat treatment at 823 K for 3 h induced the chemical reaction $4Al + 3C = Al_4C_3$. The cylinders were then hot extruded at 873 K with a 94% reduction in cross section into rods of 10 mm diameter. Material processing by ECAP was realized

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doi:10.1016/j.msea.2011.10.122

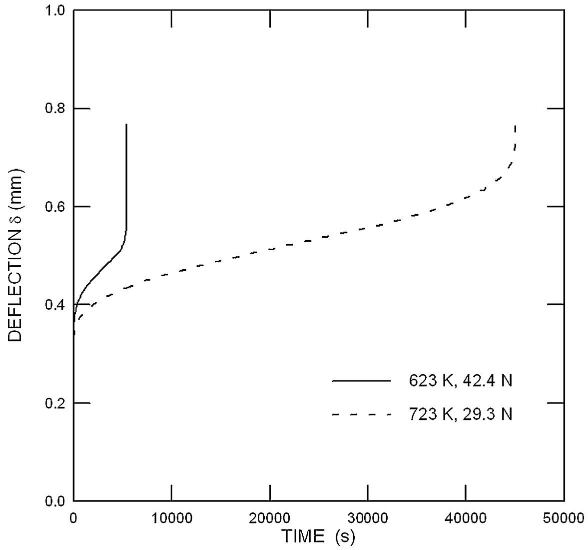

Fig. 1. Example of the time dependence of the measured central deflection.

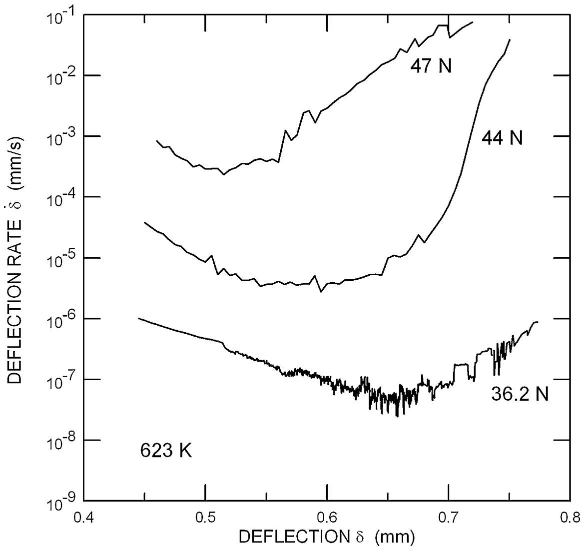

Fig. 2. Variations of the deflection rate with deflection at 623 K.

at room temperature by route C (sample rotation around the axis of about 180° after each pass), with two ECAP passes (sample sizes: diameter $D = 10$ mm and length $l = 80$ mm) and a 90° angle between the two channels.

The specimens for small punch testing were prepared by cutting slices 1.2 mm thick and 8 mm in diameter using spark erosion. The slices were perpendicular to the extrusion axis. The slices were ground carefully from both sides equally and finally polished to 1200 grit. The final thickness of 0.500 ± 0.002 mm was measured by a micrometer with a resolution of 1 μm. The small punch testing assembly is described in more details in our previous paper [17]. Particular attention was paid to geometrical features of the test. It should be expected that both the measured quantities, i.e. the minimum deflection rate and the time to fracture depend on the geometry of the test. The dimensions were selected in agreement with the Code of Practice recommended by CEN (Comité Européen de Normalisation, European Committee for Standartization) [18]: radius of spherical punch indenter $r = 1.25$ mm, lower die radius $R = 2$ mm, specimen diameter 8 mm and specimen thickness $h = 0.5$ mm. The specimen was clamped by an upper die. Central deflection was measured as the difference in the positions of the punch and lower die, using a linear variable differential transformer and was continuously recorded with a PC. The tests are performed in the constant force regime in a protective argon atmosphere. The fractures after small punch creep were observed in the electron microscope JEOL JSM 7000F.

Samples for transmission electron microscopy (TEM) were prepared as follows: experimental samples were cut by means of diamond saw to slices of a thickness from 0.3 to 0.5 mm, then mechanically ground down to a thickness of 50–70 μm and finally thinned by ion bombardment in order to obtain regions which are transparent for the electron beam. Observations were made at accelerating voltage of 100 kV.

3. Results and discussion

3.1. Small punch creep test

Examples of the dependence of the central deflection vs. time obtained in the small punch test arrangement are given in Fig. 1. It can be seen that the same general features of the curve can be observed as in conventional creep tests with clearly defined primary, secondary and tertiary stages. The curves in Fig. 2, obtained by simple numerical differentiation, emphasize that a minimum deflection rate rather than a secondary or steady-state value is reached when the decaying primary rate is surpassed by accelerating tertiary rate associated with damage processes such as cavitation, crack growth and neck formation. The dependence of the minimum deflection rate on the applied force for two temperature levels is given in Fig. 3. The dependence can be described by a power–law relationship of the form:

$$\dot{\delta}_M = A_S \cdot F^{n_S}, \qquad (1)$$

where $\dot{\delta}_M$ is the minimum deflection rate, F is the acting force and A_S is a temperature dependent constant. The values of the exponent n_S after the ECAP process: 29.0 at 623 and 23.4 at 723 K. These values are comparable with the values of the stress exponents of minimum creep rate in tension estimated for a similar Al–Al_4C_3 composite that was not subjected to the ECAP procedure [19] (21.5 at 623 K and 21.4 at 723 K, respectively).

In the tertiary stage, the deflection rate increases and finally, the fracture of the disc occurs. The dependence of the time to fracture,

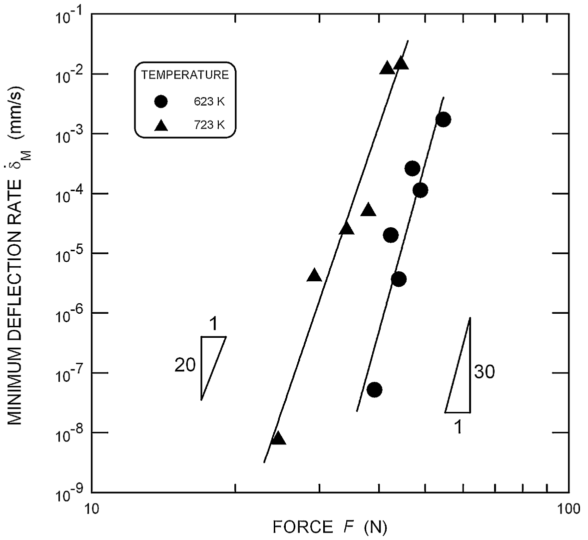

Fig. 3. Dependence of the minimum deflection rate on the applied force.