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Effects of ECAE process on microstructure and transformation behavior of TiNi shape memory alloy

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Abstract

Equal channel angular extrusion (ECAE) process was a deep pure shear deformation method for structural materials, developed in early 90s. ECAE technique has been successfully applied to several pure metals and alloys to refine the microstructure and improve the mechanical properties of materials. In the present paper, TiNi shape memory alloys have been treated by ECAE process. The effects of ECAE process on microstructures and transformation behaviors have been investigated. The initial coarse grains of TiNi alloy were refined after two passes of ECAE processes and short annealing at 1023 K. Transformation temperatures of TiNi alloy sharply decreased after two ECAE processes, then rapidly rose back when annealed at 773 K for 2 h.

Keywords: TiNi shape memory alloy; Equal channel angular extrusion; Phase transformations

1. Introduction

Ultra-fine grained materials exhibit superior mechanical properties. The equal channel angular extrusion (ECAE) technique, introduced by Segal et al. [1], has been successfully applied to produce various ultra-fine grained materials, such as low carbon steel [2,3], copper [4–6], Al alloys [7–10], pure Ti [11,12] and Ti–6Al–4V [13] et al.

However, the properties and microstructures of TiNi shape memory alloys treated by ECAE process have been reported few. Recently, Nakayama et al. [14] analyzed crystal refinement of TiNi shape memory alloy by cold rolling. Khmelevskaya et al. [15] investigated microstructure of TiNi-based shape memory alloys after severe plastic deformation. Transformation behavior of TiNi alloy after ECAE process has not been reported yet. In the present paper, the microstructure evolutions and transformation behavior of TiNi shape memory alloy after high temperature ECAE processes have been analyzed.

2. Experimental method

Experimental materials were Ti-50.6at% Ni alloy rods, with a diameter of 25 mm, after 1123 K hot forging and 773 K annealing for 2 h. Billets for ECAE process with dimensions of $10 \times 10 \times 55$ mm^3 were cut from the TiNi rod. The ECAE die was designed to yield an effective strain of ~1 by a single pass. The inner contact angle (Φ) and the arc of curvature (ψ) at the outer point of contact between channels of the die were 90° and 90°, respectively, as shown in Fig. 1. Two ECAE processes were conducted at high temperature. Keeping the die at 823 K, billets were preheated at 1123 K for 20 min before the first ECAE extrusion, and 1023 K for 20 min before the second ECAE extrusion. During ECAE process, the billet was not rotated between passages.

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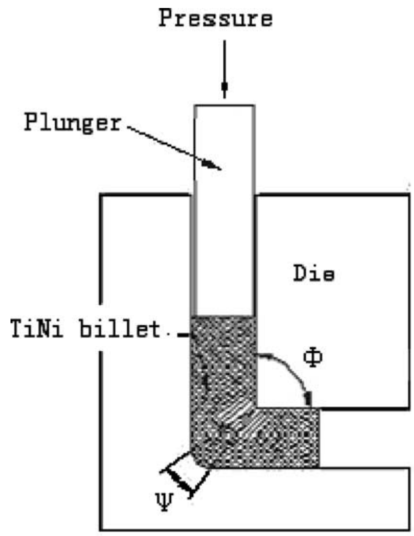

Fig. 1. Schematic illustration of the ECAE facility in the experiment.

Samples for DSC and microstructure observation were cut from billets after ECAE process along extruding direction. Some of them were subsequently annealed at 1023 K for 5–20 min to observe their microstructural changes during static re-crystallization. After mechanically polishing and eroding in a mixture solution of HF:HNO_3:H_2O with a ratio of 1:3:10, samples were observed on a NEOPHOT-1 optical microscope. Differential scanning calorimeter (DSC) measurements were carried out at heating and cooling rates of 10 K/min to analyze the phase transformation behaviors.

3. Results and discussion

3.1. Evolution of the microstructures of TiNi alloys

The initial microstructure of as-received hot forged and 773 K annealed TiNi alloy rod was shown in Fig. 2. The grains were coarse and unequal, a little extending along the axial direction of alloy rod. During the first ECAE process, billet was preheated at 1123 K for 20 min. It was found that the microstructure was coarse after the preheating.

After the first ECAE extrusion, grains were elongated in a direction parallel to the shear direction and forming an angle of ~30° respect to the extruding direction, as shown in Fig. 3(a) After the first ECAE extrusion, the billet was heated to 1023 K and held for 20 min before the second ECAE extrusion. It was observed that elongated grains after the first ECAE process became equaixed grains about 10 μm in size after 1023 K annealing for 20 min, as shown in Fig. 3(b), which revealed that there was static re-crystallization occurred during 1023 K annealing. Comparing Fig. 2 with Fig. 3(b), it was seen that TiNi grains remarkably refined after the first high temperature ECAE process and static recrystallization at 1023 K for 20 min.

Fig. 2. Optical micrograph of TiNi alloy before the first ECAE process.

Compared with the first ECAE treatment, the second ECAE extrusion resulted in denser shear bands, as seen in Fig. 3(c). Grains were elongated dramatically and parallel to the shear direction, forming an angle of ~45° respect to the extrusion direction. From Figs. 2 and 3(a)–(c), it was reasonably suggested that there were not dynamical re-crystallization occurred during high temperature ECAE processes. However, there was probably dynamical recovery occurred during high ECAE processes, especially for the first ECAE process, because these ECAE processes were performed at so high temperature and under changing temperature condition, in which the die was kept at 823 K and billets were preheated at 1123 K, 20 min and 1023 K, 20 min for the first ECAE process and the second ECAE process, respectively. Recrystallized fine grains appeared in the specimen annealed at 1023 K for 5 min after above two passages of ECAE. Elongated grains became nearly full equaixed grains when specimens were heated to 1023 K for 10–20 min, as shown in Fig. 3(d). Compared to Fig. 3(b), grains of Fig. 3(d) were finer.

Fig. 4 showed the microstructure of specimen annealed at 773 K for 2 h after above two passages of ECAE. Grains were still elongated as before, but a little bit widened. The microstructure of specimen annealed at 873 K for 2 h were similar to that in Fig. 4, which revealed that there was no re-crystallization occurred during annealing at 773 or 873 K for 2 h. It was suggested that the static re-crystallization temperature for TiNi specimen after above two high temperature ECAE processes was higher than 873 K. However, there possibly were static recoveries occurred since the elongated