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An effective approach to produce a nanocrystalline Ni–Ti shape memory alloy without severe plastic deformation

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ABSTRACT

Bulk nanocrystalline (d = 20–40 nm) Ni–Ti shape memory alloy was produced via cold marforming, followed by optimum post-heat treatment. The total accumulative strain ($\varepsilon \sim 0.7$) during the process was much lower than that typically imposed during severe plastic deformation operations (i.e., $5 \leq \varepsilon \leq 12$). In-situ heating transmission electron microscopy revealed that the present process was effective in promoting static recrystallization and limiting grain growth. The nanocrystalline alloy exhibited enhanced shape memory behaviors.

1. Introduction

Compared to their coarse-grained (CG) counterparts, nanocrystalline (NC, $d \leq 100$ nm) or ultrafine-grained (UFG, 100 nm $< d \leq 1$ μm) shape memory alloys (SMAs) have many advantages, such as better balance between strength and ductility, enhanced thermal cyclic stability, and higher recovery stress [1,2]. The well-known processes to obtain such extremely fine grains include severe marforming [3,4] or severe ausforming [4,5], utilizing equal-channel angular extrusion (ECAE), and high pressure torsion (HPT). For example, disc-type 50.6Ni–49.4Ti (atomic percent, hereafter), consisting of amorphous and NC structures (d = 5–10 nm), have been fabricated via HPT [1], while rod-type 50.6Ni–49.4Ti and 49.8Ni–42.2Ti–8Hf, having UFG structures (d = 200–500 nm), have been produced via ECAE [1,2]. High-strain ε values of about 5–12 or elevated temperature conditions (400–650 °C) were required to complete grain refining in such manners. For widespread application of NC/UFG SMAs, a reduction in the strain required to achieve NC/UFG structures would be beneficial because conventional metal-forming processes, such as rolling, extrusion, drawing, and their concomitant high strain rate $\dot{\varepsilon}$ could be used.

In this regard, the authors' have been attracted by some interesting and controversial results. For example, studies indicate that NC or UFG Ni–Ti SMA can be fabricated by utilizing cold rolling followed by static heat treatment at 400–500 °C [6–8]; however, systematic analysis on such interesting phenomena have been somewhat limited, and no effective strategy to obtain NC/UFG SMA has been suggested as yet. By contrast, according to independent research [9,10], CG ($d > 10$ μm) structures were found to be *not* fully refined even after a very similar thermomechanical process to the abovementioned (cold rolling + static heat treatment at 400–500 °C) for the same alloy. Such controversial results might be attributed to two causes: (1) different starting phases, i.e., austenite or martensite, which are greatly affected by the real chemical composition, testing environment, and deformation heating, and (2) different final annealing temperatures, which can give rise to different static recrystallization kinetics. If the effects of these processing conditions on the final grain size are made clearer, an effective method may be developed to produce NC/UFG SMAs without severe plastic deformation.

Thus, the objectives of the present work were as follows: (1)

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investigate the effect of the starting phase prior to deformation as well as the post-annealing temperatures on final microstructures; (2) establish a more effective and systematic process to reduce the grain size of SMAs; and (3) assess the feasibility of forming an NC structure in SMAs using lower ε values than those in prior investigations.

2. Material and methods

The material used in this study was 50.4Ni–49.6Ti SMA prepared by hot forging at the temperature of ~950 °C subsequent to vacuum arc remelting; the thickness of forged billet was ~25 mm. Finally, it was solution heat treated at 850 °C for 2 h and quenched in water; the prior austenite grain size was ~120 μm (Fig. 1). The martensite transformation start (M_s) and finish (M_f) temperatures were 29 and 14 °C, respectively, while the reverse transformation start (A_s) and finish (A_f) temperatures were 40 and 58 °C, respectively.

To investigate the effect of stable phase prior to cold rolling on the as-rolled microstructure, rod-type samples (Ø: 6.6 mm × L: 110 mm) were machined and then groove rolled using either (1) cold ausforming at 200 °C ($>A_f$) or (2) cold marforming at −5 °C ($<M_f$) to a total of $\varepsilon \sim 0.7$ with inter-pass heating or chilling. In other words, the former process started with an *austenite* phase, while the latter started with a *martensite* phase. The homologous temperatures for both processes (0.29 and 0.17 T_m) were low enough to maintain cold rolling conditions. Subsequent to the both processes, the samples were annealed at temperatures of 400–600 °C for 1 h to examine the effect of post-heating temperatures on static recrystallization behavior.

Differential scanning calorimetry (DSC) measurements were made at a heating and cooling rate of 0.17 K/s using a TA Instrument DSC-2010. Thin foils for TEM were prepared via twin-jet electropolishing at −35 °C using a mixture of 7.5% perchloric acid and 92.5% methyl alcohol. In-situ heating TEM at 300 °C ($>A_f$) was utilized using a JEM-2100 to examine the austenite grain size. Tensile tests were performed using an INSTRON 5585H machine with an advanced video extensometer; the gauge length was 30 mm. Except for in-situ TEM, all the experiments were repeated independently more than three times and the results were consistent.

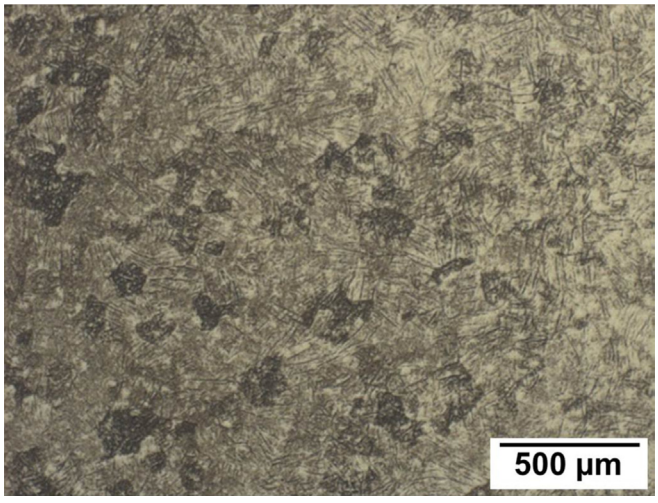


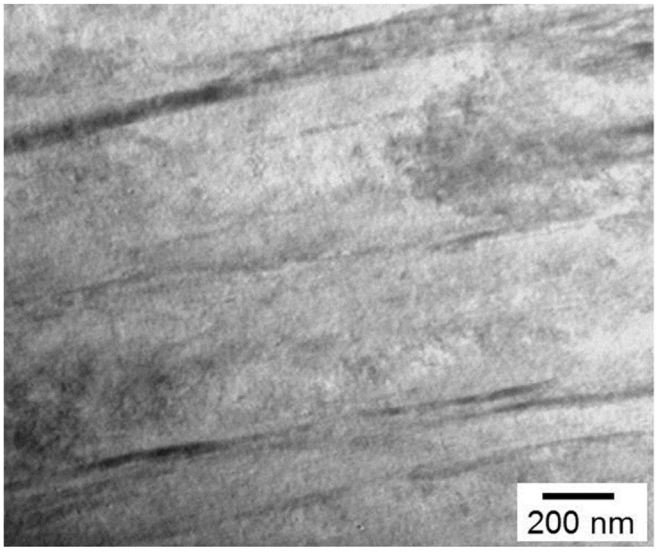
Fig. 1. Optical micrograph for the sample heat-treated at 850 °C for 2 h followed by water quenching.

3. Results and discussion

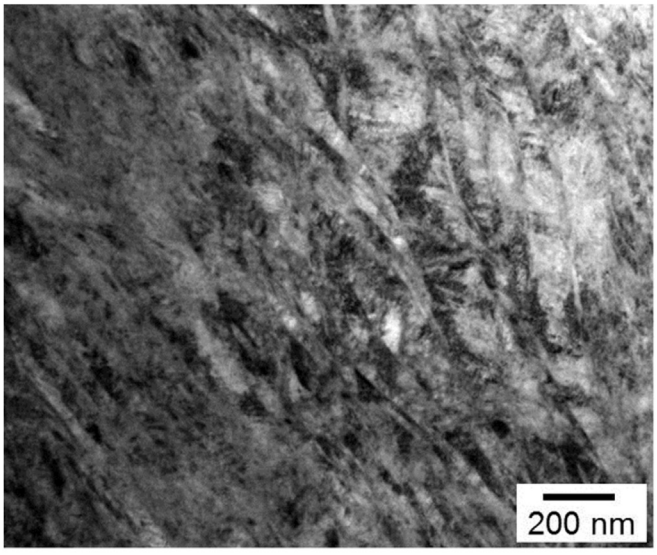
3.1. Microstructures

The microstructures of the as-deformed samples revealed noticeable differences in terms of their morphology and substructure. For the cold ausforming sample, local dislocations in the less distorted matrix were observed (Fig. 2a); for the cold marforming sample, highly distorted and fragmented laths with a high dislocation density, as evidenced by a contrast change in the TEM image, were formed (Fig. 2b).

For the cold ausforming sample followed by annealing at 400 °C, the microstructure was partially recrystallized, and grains as large as several micrometers were found to exist with some nano-sized grains (Fig. 3a). However, for the cold marforming sample, the same heat treatment gave rise to an almost fully recrystallized



(a)



(b)

Fig. 2. Room temperature TEM images of the microstructures developed after (a) cold ausforming and (b) cold marforming prior to subsequent annealing.