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Fabrication of bulk nanocrystalline Fe–Ni alloys with high strength and high ductility by an electrodeposition

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ABSTRACT

The tensile behavior of electrodeposited bulk nanocrystalline Fe–Ni alloys with a grain size of approximately 15 nm was investigated. Electrodeposited alloys with a nickel content of 43–56 wt% have a single-phase face-centered cubic structure. These alloys exhibited a good combination of tensile strength and ductility in tensile tests at room temperature. In particular, bulk nanocrystalline Fe–44 wt% Ni exhibited the high tensile ductility of 1.7 GPa and significant ductility of 16%, and the tensile specimen after fracture showed clear necking. Fracture surface observation indicated that dimples, the size of which was 10 times the grain size, developed in electrodeposits with high ductility of above 10%. The results of this study point out that grain clusters can have a role in plastic deformation during tensile loading.

1. Introduction

Extensive efforts have been made to explore the mechanical properties of nanocrystalline metals with grain sizes less than 100 nm [1,2]. Polycrystalline metal strength drastically increases as the grain size reaches the nanometer range, following a Hall–Petch relationship [3,4]. Moreover, molecular dynamics (MD) simulations [5,6] suggest that the deformation mechanism changes from dislocation activity to grain boundary activity, i.e., grain boundary sliding [7], grain boundary migration [8], and grain boundary dislocation emission [9], at a critical grain size. The critical grain size is usually observed in the range of 10–20 nm for face-centered cubic (fcc) metals [10]. Nanocrystalline metals are expected to have potential in a variety of new industrial applications [2]. However, the practical use of nanocrystalline materials is limited, because most of them exhibit low tensile ductility [11–14].

Recent experimental studies [15–19] have reported newly developed fabrication processes that result in fcc structure nanocrystalline metals with good ductility. In fact, Li and Ebrahimi [16] electrodeposited nanocrystalline Ni–Fe alloys with a high strength of 2.4 GPa and a tensile elongation of 6%, and they mentioned that nanocrystalline metals are not intrinsically brittle. Brook et al. [17] investigated the effect of specimens' thickness on tensile ductility in electrodeposited nanocrystalline Ni–Fe alloys. The results of their detailed study indicated that limited sample thickness ($< 100\ \mu m$) could suppress tensile ductility. Based on these results, we suggest that improvement in the electrodeposition process allows for the production of idealized bulk specimens that can be used to understand tensile behavior in nanocrystalline metals. To develop an experimental understanding of typical deformation mechanisms in bulk nanocrystalline metals, high-quality specimens, which have a high tensile ductility above 10%, with a grain size range of 10–20 nm are required. Therefore, this study aims to optimize the electrodeposition process for producing high ductility bulk nanocrystalline Fe–Ni alloys with a grain size of approximately 15 nm.

2. Experimental procedure

Four bulk samples of nanocrystalline Fe–Ni alloys, with varying nickel contents (43, 44, 46, and 56 wt%), were electrodeposited by adjusting the iron sulfate and nickel sulfamate concentration. In this study, the following labels were assigned to electrodeposited Fe–Ni alloys with different nickel contents: Fe–43Ni, Fe–44Ni, Fe–46Ni, and Fe–56Ni. The details of the bath compositions for bulk nanocrystalline Fe–Ni alloys are given in Table 1. All samples were deposited on copper substrates of commercial purity using two counter electrodes of titanium baskets with iron plates (99.8%) and nickel plates (99.98%). The main components of the electrodeposition system are described in our previous study [19]. All electrodepositions were run at a current density of 10 mA/cm^2, a bath temperature of 50.0 ± 0.5 °C, and a pH of 2.3 ± 0.1. The pH value of the solutions was maintained by adding drops of either 1.0 mol/L sulfamic acid or 5.0 mol/L sodium hydroxide.

The nickel content of the electrodeposits was determined by energy-dispersive X-ray spectrometry analysis using a scanning electron microscope (HITACHI S-4800). The carbon content of the

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electrodeposits was quantified by an IR absorption method after combustion in a high-frequency induction furnace. X-ray diffraction (XRD, RIGAKU Ultimate IV) analysis was performed using Cu Kα radiation to estimate grain size. The microstructure was observed using a transmission electron microscope (TEM, JEOL JEM-2100F) operated at 200 kV. For a tensile test, dog-bone specimens with a gauge length of 12 mm, width of 4.0 mm, and thickness of approximately 0.7–0.8 mm were machined by electrical discharge machining from the as-deposited plates, and the copper substrate was removed by mechanical polishing. Tensile tests were performed at a strain rate of $1 \times 10^{-3}\ \mathrm{s}^{-1}$ and room temperature. The plastic deformation of the specimen after fracture was measured by the change in the gauge length. Each reported data point for tensile properties represents the average of three measurements.

3. Results and discussion

Four bulk samples with a single-phase fcc structure were prepared by electrodeposition from Baths A–D. Fig. 1 shows bright-field TEM micrographs of the planar view of the electrodeposited bulk nanocrystalline Fe–Ni alloys. Each sample had a microstructure with a grain size of approximately 10–20 nm. The grain sizes of Fe–56Ni, Fe–46Ni, Fe–44Ni, and Fe–43Ni estimated using the XRD peak width and Scherrer's equation were 15, 15, 14, and 14 nm, respectively. The calculated grain size of each sample was comparable with the sizes observed in TEM images.

The stress–strain curves of electrodeposited bulk nanocrystalline Fe–Ni alloys obtained from tensile tests at room temperature are shown in Fig. 2. The ultimate tensile strength and plastic deformation were determined by measuring the change in the gauge length before and after fracture. Tensile strength of Fe–56Ni,

Table 1
Bath compositions (in g/L) for bulk nanocrystalline Fe–Ni alloys.

Chemicals	Bath A	Bath B	Bath C	Bath D
Iron sulfate	70.0	100.0	105.0	110.0
Nickel sulfamate	215.0	180.0	175.0	170.0
Nickel chloride	20.0	20.0	20.0	20.0
Boric acid	40.0	40.0	40.0	40.0
Saccharin sodium	5.0	5.0	5.0	5.0
Sodium lauryl sulfate	0.3	0.3	0.3	0.3

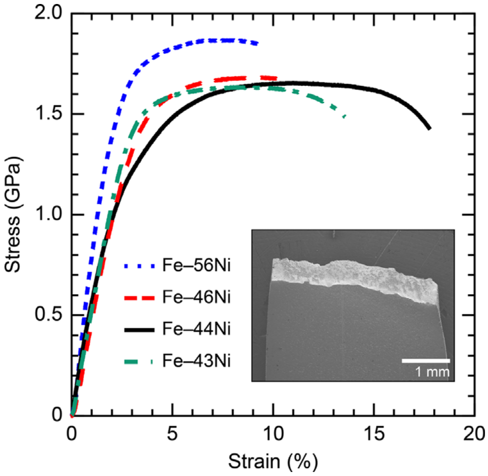

Fig. 2. Stress–strain curves of electrodeposited bulk nanocrystalline Fe–Ni alloys. The inset image shows the gauge section of the actual Fe–44Ni specimen after fracture.

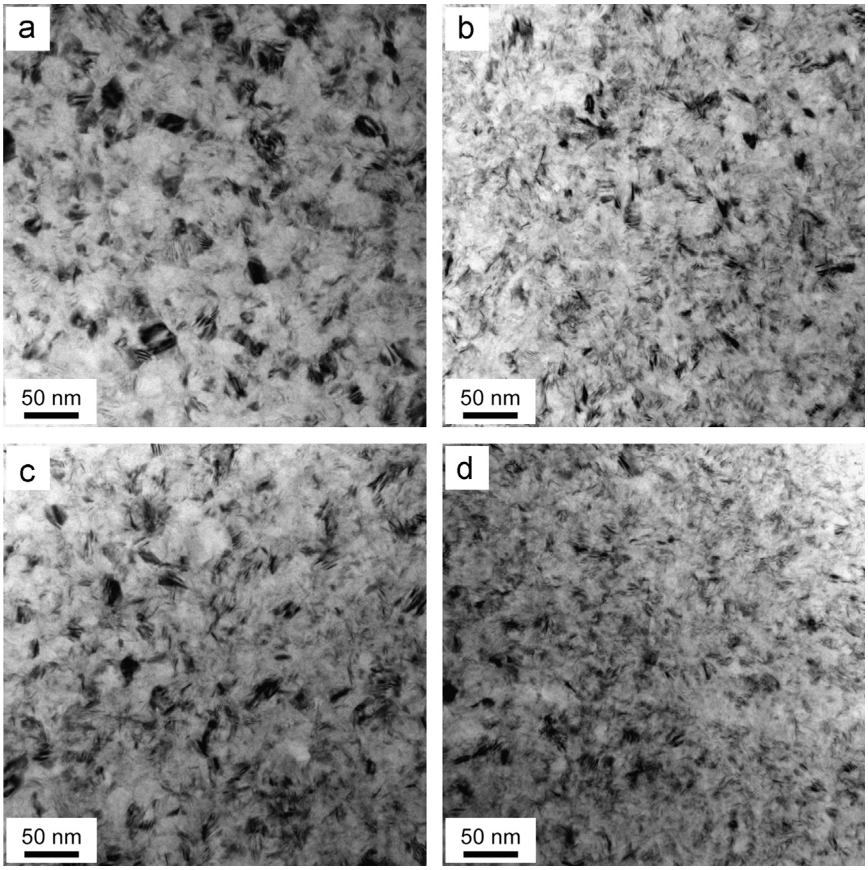

Fig. 1. Bright-field TEM images showing the planar views of electrodeposited bulk nanocrystalline Fe–Ni alloys: (a) Fe–56Ni, (b) Fe–46Ni, (c) Fe–44Ni, and (d) Fe–43Ni.