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Formation of high-strength nano-micro duplex structures in electrodeposited Ni-based alloys

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Abstract

Nanocrystalline Ni–W electrodeposits with finely dispersed micrometer sized array holes have been prepared using the UV-lithographic technologies. Tensile stress and the total elongation at fracture of the monolithic Ni–16 at.% W nanocrystalline alloys in the grain size range of 5–10 nm attained to 1700 MPa and about 1%, respectively, and that of the Ni–W nanocrystalline alloys with finely dispersed array holes having diameter of 30 μm with regular intervals of 100 μm increased to maximum values of 2200 MPa and about 1.2%, respectively.

Keywords: Electrodeposition; Ni–W; Nano-micro duplex structure; UV-lithographic; Tensile strength; Plastic deformation

1. Introduction

Electrodeposition is an excellent technique for producing nanocrystalline materials. In our previous study, we developed an aqueous plating bath for producing nanocrystalline Ni–W alloys having high hardness and good bending deformability [1]. However, use of these nanocrystalline alloys as structural components in practical application is limited because of their severe brittleness in tensile testing mode. Similar failure has also been observed in monolithic glassy metals, and an icosahedral phases [2]. A ductile bcc phase [3] distributed in a glassy matrix significantly improved their deformation behavior. However, this structural-control technique is not generally applicable in nanocrystalline materials.

In the present study, nanocrystalline Ni–W electrodeposits with finely dispersed micrometer sized array holes have been prepared using the UV-lithographic technologies to control the initiation and the propagation of the shear bands during tensile testing.

2. Experimental procedures

The plating bath for the deposition of Ni–W nanocryastalline alloys is an aqueous solution of nickel sulfate and sodium tungstate together with complexing agents. Trisodium citrate and ammonium chloride were introduced to form complexes with both Ni and W in the plating bath solution. The composition of the bath and other details of the electrodeposition process are given elsewhere [4,5]. Fig. 1 shows a schematic diagram of the tensile test specimens. Ni–W electrodeposition was done on Cu sheets as substrates for various shapes of tensile test specimen prepared by UV-lithographic technologies. The electrodeposited Ni–W films were separated from the Cu substrate by immersing the samples in an aqueous solution containing CrO_3 and H_2SO_4. The tensile test was carried out for the electrodeposited films having a thickness of about 20 μm and gage length width of 5 mm and 2 mm by using an Instron-type machine with a strain rate of $3.67 \times 10^{-4}\ s^{-1}$. The sample with finely dispersed array holes of 20 μm and 30 μm in diameter with regular intervals of 100 μm were prepared. Elemental concentrations of the electrodeposits were analyzed by wavelength dispersive X-ray spectroscopy (WDS) in a scanning electron microscope. Structure of the electrodeposited alloys was examined by X-ray diffraction using Cu K_α radiation (40 kV to 30 mA). Vickers microhardness was measured with 0.2 kg load and loading time of 15 s in cross section.

3. Results and discussion

Fig. 2 shows the X-ray diffraction pattern of as-electrodeposited Ni–16.3 at.% W alloy. The diffraction pattern reveals broadened fcc-peaks from the Ni–W solid solution. These electrodeposits had nanocrystalline structure and their grain size is of about 7 nm in diameter, as obtained by the

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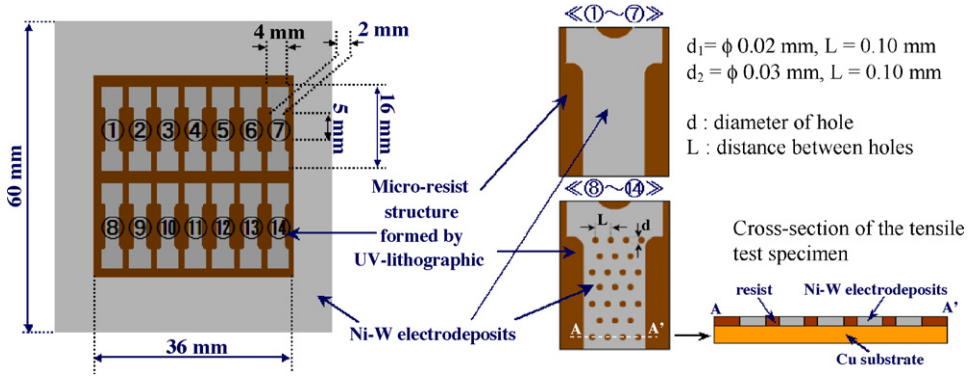

Fig. 1. Schematic drawing of the tensile test specimens of Ni–W nanocrystalline alloys.

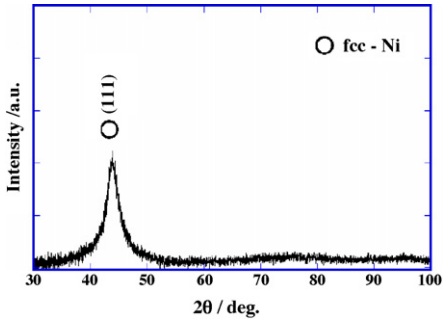

Fig. 2. X-ray diffraction pattern of the Ni–16.3 at.% W electrodeposit.

Scherrer formula for the fcc (1 1 1) diffraction peak. Vickers microhardness of HV660 for the as-electrodeposited Ni–W alloys was a very large value as compared with HV200 for the pure-Ni electrodeposits.

Fig. 3 shows the high-resolution TEM image of the as-electrodeposited Ni–16.6 at.% W alloys. Grain sizes of about 5–10 nm are observed. These grain sizes are consistent with that obtained from the X-ray analysis.

Fig. 4 shows the stress–strain curves obtained by tensile testing of the monolithic Ni–W nanocrystalline sample, the 20 μm array holes-sample and the 30 μm array holes-sample. In the case of the monolithic sample, the apparent yield stress, the tensile strength and the total elongation at fracture were 1000 MPa, 1700 MPa and about 1%, respectively. With finely

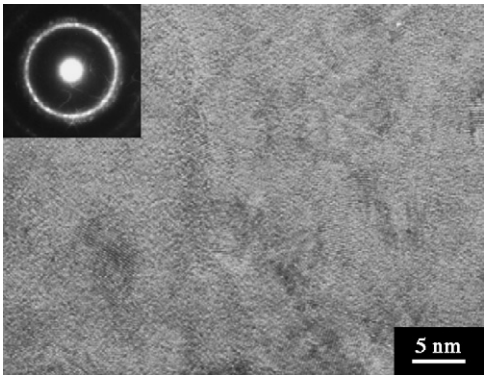

Fig. 3. High-resolution TEM image and the corresponding selected area diffraction pattern of the Ni–16.6 at.% W alloy.

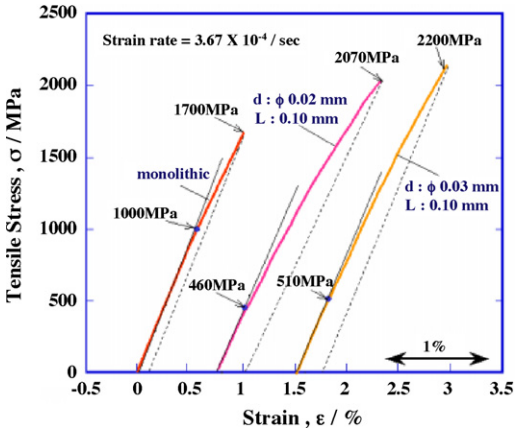

Fig. 4. Stress–strain curves obtained by tensile testing of the monolithic sample, the 20 μm array holes-sample, and the 30 μm array holes-sample. *d*: diameter of hole; *L*: distance between holes.

dispersed array holes in the Ni–W nanocrystalline alloys, the apparent yield stress of the electrodeposits decreased drastically to 460 MPa, while the tensile strength and the total elongation at fracture increased to 2070 MPa and 1.2% for the 20 μm array holes-sample, and 2200 MPa and about 1.2% for the 30 μm array holes-sample, respectively.

Fig. 5 shows optical micrographs of the surface morphology near the fracture surface of the tensile specimens. In the case of the monolithic sample, shear bands have been observed only near the fracture surface, while in the case of the 20 μm array holes-sample, many shear bands have been observed in wide area within about 320 μm from the fracture surface, resulting in a large increase of the plastic deformation during the tensile test.

Table 1 shows some typical results of the tensile tests for the monolithic and samples with array of holes. The stress concentration factors for 20 μm and 30 μm holes array are 2.45 and 2.15, respectively. The decrease of the apparent yield stress can be roughly explained by the stress concentration factors of these array of holes. On the other hand, the tensile stress at fracture increased up to 2200 MPa with the 30 μm array of holes, and the total elongation at fracture also increased up to about 1.2%. In this nanocrystalline alloy, there might be no work hardening effects during the tensile test, so the increase of the tensile stress with their plastic deformation above the apparent yield stress

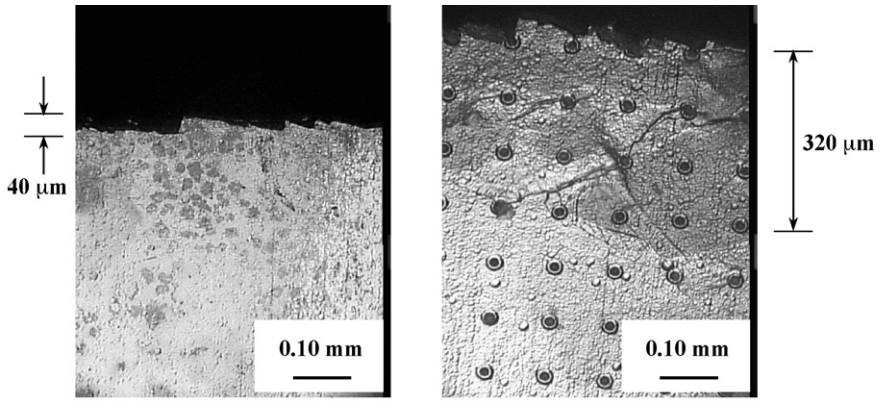

Fig. 5. Optical micrographs of the surface morphology near the fractured surface of the tensile specimens.

Table 1
Some typical parameters of the tensile tests for the monolithic and samples the array of holes

	The monolithic sample	The 20 μm array holes-sample	The 30 μm array holes-sample
The tensile stress at fracture (MPa)	1700	2070	2200
The apparent yield stress (MPa)	1000 (σ_m)	460 (σ_{20})	510 (σ_{30})
The elongation at fracture (%)	1.0	1.2	1.2
The elongation of plastic deformation (%)	0.10	0.24	0.17
Stress concentration factor	1.0	2.45	2.15
σ_m/σ_{20}, σ_m/σ_{30}	1.0	2.17	1.96
Vickers hardness (H_v)	660		

may be due to multiple localized yielding to form shear bands around the finely dispersed holes in wide area. These results suggested that the true yield stress of the Ni–W nanocrystalline alloy may be more than 2200 MPa.

4. Conclusions

In the case of the monolithic Ni–W nanocrystalline samples, the apparent yield stress, the tensile strength and the total elongation at fracture were 1000 MPa, 1700 MPa and 1%, respectively. With finely dispersed holes with diameters of 20 μm and 30 μm in the Ni–W alloys, the apparent yield stress decreased drastically, while the tensile strength and the total elongation at fracture increased up to 2200 MPa and 1.2%, respectively. The decrease of the apparent yield stress can be roughly explained by the stress concentration factors of these array holes, while the increase of the tensile stress at fracture may be due to the multiple yielding to form shear bands around the finely dispersed holes during tensile test.

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