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### Short communication

## Effect of electropulsing treatment on microstructure and tensile fracture behavior of nanocrystalline Ni foil



MATERIALS<br>SCIENCE & **ENGINEERING** 

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#### **ABSTRACT**

Electropulsing treatment (EPT) is utilized to increase the plasticity of nanocrystalline Ni foil. Uniaxial tensile tests at room temperature are adopted to investigate the influence of EPT on the mechanical properties under different peak current density. It is shown that electropulsing with certain strength can improve the elongation significantly. The evolution of the fracture morphology, stacking faults, twinning and dislocation of nanocrystalline Ni under EPT is studied via scanning electron microscopy (SEM) and transmission electron microscopy (TEM), and the plastic deformation mechanism under EPT is also researched.

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#### 1. Introduction

Microforming technology using nanocrystalline foil is one of the most effective ways to resolve the problem of manufacturing complex shell parts at a low cost and in high efficiency in MEMS, which has attracted worldwide attention  $[1-4]$  $[1-4]$ . Generally speaking, some inferior properties of metallic nanocrystalline material such as poor plasticity and poor formability at room temperature will restrict its application greatly. Therefore, lots of researches on how to improve the plasticity and formability of nanocrystalline foil have been carried out. Matsui et al. [\[5](#page--1-0),[6\]](#page--1-0) reported that the orientation and additives are important to improve the tensile ductility of electrodeposited bulk nanocrystalline Ni–W alloys. Lian et al. [\[7](#page--1-0),[8\]](#page--1-0) indicated that broad grain size distribution and dislocation activity are suggested as being responsible for the enhanced ductility of nanocrystalline Ni. In addition, from the perspective of forming process, some extra process measures could also be taken to solve the above mentioned issues.

Previous work indicated that electropulsing treatment (EPT) has a prominent impact on the microstructure and the mechanical properties of metallic materials [\[9](#page--1-0)–[14\]](#page--1-0). Conrad et al. [\[15\]](#page--1-0) proposed that the EPT can promote the superplasticity of 7474 aluminum alloy. Tang et al. [\[16,17\]](#page--1-0) showed that EPT was an efficient approach for accelerating dynamic recrystallization and significantly increased the elongation at a high strain rate of AZ31 Mg alloy.

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<http://dx.doi.org/10.1016/j.msea.2016.01.075> 0921-5093/© 2016 Elsevier B.V. All rights reserved. However, the current studies are mostly focusing on macrostructural materials while less work has been done on the effect of EPT on metallic nanocrystalline foil. Therefore, high-density EPT is applied to improve the plasticity of the nanocrystalline foil during this research. The effect of EPT on mechanical properties and microstructure of the nanocrystalline Ni foil at room temperature is studied and the plastic deformation mechanism of nanocrystalline foil under EPT is also analyzed.

#### 2. Material and methods

The electrodeposited nanocrystalline Ni foils with a thickness of 50 μm are prepared in this experiment. The electrodeposition bath and conditions are set as follows: A nickel sulfamate bath (the value of pH is 3), containing 15 g/L nickel chloride, 300 g/L nickel sulfate, 30 g/L boric acid and 1 g/L saccharin is used to produce Ni foils. Square waves with a duty cycle 50% and peak current density up to  $2$  A/dm<sup>2</sup> are employed during pulse electro-deposition. The deposition temperature is 323 K. A nickel plate of 99.99% purity is used as the anode and a stainless steel plate is used as the cathode. [Fig. 1](#page-1-0) shows the TEM bright field image of the electrodeposited nanocrystalline Ni. As shown in [Fig. 1](#page-1-0), the average grain size of the nanocrystalline Ni is approximately 100 nm. Uniaxial tensile tests are performed on an INSTRON-3340 universal testing machine, using the sample of 3 mm width and 10 mm gauge length. The change of gauge length is experimentally measured and calculated the plastic elongation. The tests are carried out at a rate of

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Fig. 1. TEM bright field image (a) and grain size distribution (b) for electrodeposited nanocrystalline Ni.



Fig. 2. The schematic view of EPT.

0.075 mm/min. The dynamic electropulsing is performed on the sample being tensile deformed. The EPT process is schematically shown in Fig. 2. A pulse power supply is applied to discharge multiple pulses with various current densities. As shown in Fig. 2, the form of the pulse current is DC square wave with very high amplitude (maximum value is 345 A) and low duty ratio (duty ratio  $D = t_i/t_p$ , minimum value is 0.1%). Such a waveform could generate very high peak current and relatively low root-meansquare current, which not only help to improve the plasticity of the material, but also avoid the heating of the samples. Multiple electropulses are applied on two electric contactors with a distance of 20 mm. Current parameters, including duty ratio  $(D=0.1\%)$  and peak current density  $(J=0, 0.6 \times 10^3, 1.3 \times 10^3,$  $1.65 \times 10^3$ ,  $1.85 \times 10^3$ ,  $2.3 \times 10^3$  A/mm<sup>2</sup>) are controlled by the pulse power supply. Due to the small root-mean-square current density  $(J_m=0, 0.6, 1.3, 1.65, 1.85, 2.3$  A/mm<sup>2</sup>) and the short duration time of the tests ( $< 600 s$ ), the surface temperature of the samples would not increase too much ( $<$ 323 K). Six specimen groups are designed and three duplicate specimens are arranged in each group. Tensile fracture is examined by HITACHI S-4800 scanning electron microscope and microstructure is observed by CM-12 transmission electron microscopy.

#### 3. Results and discussion

The results of the EPT and non-EPT samples based on uniaxial tensile tests at room temperature are shown in [Fig. 3](#page--1-0). As shown in [Fig. 3](#page--1-0)(b), the elongation of the EPT samples has been improved properly comparing with the non-EPT samples, and also with the increasing of electropulsing current density, the sample elongation increases gradually. But with the increasing of pulse current density, the elongation raises nonlinearly while the tensile strength inclines to decrease. As shown in Fig.  $3(b)$ , the elongation curve could be roughly divided into three stages. (1) under the conditions of the small current density  $(0.6 \times 10^2 \text{ A/mm}^2)$  $1.3 \times 10^3$  A/mm<sup>2</sup>), the elongation increases moderately, while the intensity decreases slightly; (2) with the current density increasing  $(1.65 \times 10^3 \text{ A/mm}^2)$ , the curve slope becomes smaller, while the intensity of the sample suddenly increases reversely; (3) while

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