



Optimal microstructural design for high thermal stability of pure FCC metals based on studying effect of twin boundaries character and network of grain boundaries

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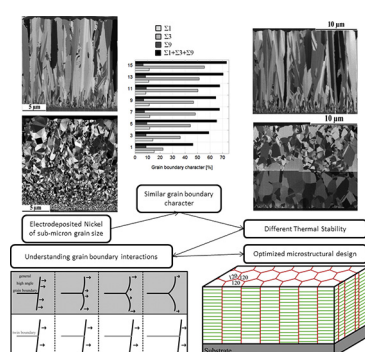
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HIGHLIGHTS

- Three nickel layers of comparable grain size and twin boundaries density, but different connectivity are electrodeposited.
- Incoherent twin boundaries density and grain boundaries interaction are dominant factors influencing the thermal stability.
- An optimal microstructural design to maximize thermal stability of materials with sub-micron grain size is proposed.

GRAPHICAL ABSTRACT



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ABSTRACT

Three nickel electrodeposits with comparable grain size were synthesized by tailoring the electrodeposition conditions. Thorough microstructural characterizations including electron backscatter diffraction, ion channeling contrast imaging, electron channeling contrast imaging, transmission Kikuchi diffraction, transmission electron and high annular dark-field imaging were applied. The deposits contain a high density of twin boundaries with similar microstructures in terms of grain boundary character. These materials were annealed at various temperatures to study the microstructural evolution, and hence, their thermal stability. The differences in the character of twin boundaries and morphology of the grain boundaries in as-deposited state and their influence on the microstructural evolution at elevated temperatures are analyzed. The importance of incoherent twin boundaries, and the interaction of mobile general high angle boundaries with stationary boundaries are discussed. Finally, an optimal design for high thermal stability is proposed, based on the mechanisms that were inferred from the results.

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1. Introduction

More than a half of the overall nickel produced worldwide is used in the form of coatings and nickel electrodeposition is the most common

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process to coat nickel [1]. High wear resistance, relatively high hardness, barrier properties, corrosion resistance, among others makes nickel electrodeposition of practical importance for a large range of applications [1]. Functional, protective and decorative nickel coatings have been applied in industry [1]. Since thermal stability determines the maximum service time/temperature, it is of high importance for many applications and hence, there is a large body of research on this subject [2–8]. The excess free energy of grain boundaries, grain boundary junctions etc. is the major driving force for grain growth in materials [9–11]. In order to stabilize the microstructure of nickel electrodeposits, very often alloying with other elements is employed [6,12–18]. The main mechanisms for stabilization is (i) kinetic and (ii) thermodynamic [19–21]. In the kinetic approach, the grain boundary mobility is reduced (e.g. solute drag on the boundary motion). In the thermodynamic approach, the driving force for grain growth is reduced by incorporation of a high fraction of low energy boundaries in the microstructure [19]. However, stabilization of pure material has gained interest in recent years [22,23]. Coherent twin boundaries (CTBs) in face centered cubic (FCC) materials are known to have low mobility [24] combined with low stored energy [25]. Thus, a high density of CTBs in the microstructure should improve the thermal stability of a material [26] due to kinetic and thermodynamic reasons. This has been empirically demonstrated to apply for a nano-twinned microstructure with a high density of CTBs, where improved thermal stability was observed as compared to a nanocrystalline microstructure of equal average crystallite size, but without twins [27,28]. In addition to the boundary character, the network of grain boundaries plays an important role in the material properties as emphasized in the concept of grain boundary engineering (GBE) [29–32]. It is therefore suggested that a GBE microstructure with an abundance of CTBs, is potentially of high thermal stability. This was demonstrated for nickel deposits, where, among others, multi-twinned grains were observed to have the highest thermal stability [33,34].

In the present work, it is attempted to elucidate on the mechanism (s) governing the thermal stability of pure FCC nickel electrodeposits with sub-micron grain size and high density of CTBs; and ultimately outline an optimal microstructure to achieve high thermal stability and mechanical strength. To this end, three different nickel electrodeposits with comparable microstructures in terms of grain size and grain boundary character distribution are synthesized and thoroughly studied. Based on the experimental results, the underlying mechanisms of microstructural evolution are identified and an optimal microstructural design for high thermal stability of pure FCC metals is proposed.

2. Experimental

2.1. Materials

Electrochemical deposition of nickel layers was carried out from a Watts-type electrolyte with the following chemical composition: 300 g·dm⁻³ nickel sulfate NiSO₄·7H₂O, 35 g·dm⁻³ nickel chloride NiCl₂·6H₂O, 40 g·dm⁻³ boric acid H₃BO₃. Nickel was electrodeposited from the electrolyte onto amorphous Ni-P substrates for three different combinations of pH and current density (see Table 1). The pH of the electrolyte was adjusted to the intended value by addition of sulfuric acid or ammonia. Further details of the synthesis parameters are provided elsewhere [35,36].

To study the thermal stability of the material, the as-deposited samples were cut into smaller pieces of 10 × 5 mm². They were annealed for

30 min at 473, 573, 673 and 873 K followed by cooling in air. Annealing at 473 K and 573 K was performed in air while argon was used for annealing at 673 K and 873 K, as at higher temperatures oxidation can be more pronounced. Never the less, it was verified that annealing in air and argon for the present temperature and time ranges has the same influence on the grain growth. The annealed samples are named with a postfix of temperature to the as-deposited sample name. For instance, S1-473 refers to sample S1, annealed at 473 K.

It is worth mentioning that the annealing temperature range of 473–873 K corresponds to homologous temperature range of 0.27–0.50 for nickel. This homologues temperature range is chosen as for iron and nickel below 0.3 the microstructural evolution is very limited and substantial grain growth takes place at 0.4 [37].

2.2. Sample preparation for microscopy

Electron backscatter diffraction (EBSD) and electron/ion channeling contrast imaging (ECCI/ICCI) were used to investigate the entire cross-sections in each deposit. This enabled to characterize the evolution of the microstructure along the growth direction (GD). Cross-sections of the samples for scanning electron and ion microscopy were prepared by grinding on SiC paper of grade 1000 and 4000, followed by mechanical polishing with 3 µm and 1 µm diamond suspension, followed by mechanical-chemical polishing with 0.04 µm colloidal silica. To ensure that the deformed layer introduced by mechanical polishing was entirely removed, the samples were milled with a focused ion beam (FIB) of Ga⁺ ions at 30 kV in an FEI Helios NanoLab™ 600 dual beam microscope. FIB milling was carried out in two steps: firstly about 700 nm were removed applying a current of 2.8 nA (rough milling), and then an additional 50 nm were removed by milling with a current of 0.46 nA (gentle milling). This sample preparation provided an artifact-free surface that is suitable for surface sensitive characterization techniques such as EBSD [38].

For transmission electron microscopy, an electron transparent foil was prepared in a FEI Helios Nanolab™ 600 dual beam microscope, using an in-situ lift-out technique [39], in which 30 kV Ga⁺ were used for the penultimate step. For the final step (removal of amorphous layer), 2 kV Ga⁺ with a current of 6 pA was applied with an incident angle of 7° to both sides of the foil for 10 min. The foil covers the whole thickness of the deposit and contains the GD within the plane of the foil.

2.3. EBSD

2.3.1. Data acquisition

To examine the character of the grain boundaries and their connectivity, orientation mapping was performed in an FEI Helios NanoLab™ 600 equipped with an EDAX-TSL EBSD system. The EBSD measurements were carried out on a hexagonal grid with an electron probe current of 5.5 nA at an acceleration voltage of 12 kV and a step size of 25 nm.

2.3.2. Data analysis

The OIM 6™ software was used for the analysis of the acquired EBSD data. Post-acquisition treatment of the recorded EBSD data included the following cleaning steps using routines available in the OIM 6™ software. Firstly, the confidence index (CI) [40] of every point in the map within a recognized grain was assigned to the highest confidence index CI value found in that grain (a grain was defined as a region consisting of at least three connected points with misorientation of <5°). Secondly, by so-called grain dilation, a point with an orientation that does not belong to any grain is assigned the orientation of the majority of neighboring points. Afterwards, all data points with CI below 0.1 were disregarded. It is noted that the cleaning procedure mainly discards the measured data points outside the deposit area (substrate and beyond the top surface) and has a very limited effect on the actual area

Table 1
Sample names and corresponding electrodeposition conditions.

Sample name	pH	Current density [A dm ⁻²]
S1	4.5	2
S2	4.5	5
S3	2.0	10

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