Materials Characterization 130 (2017) 173–180

Contents lists available at ScienceDirect

Materials Characterization

journal homepage: www.elsevier.com/locate/matchar

# Characterization of high strength nickel thin sheets fabricated by differential speed rolling method

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ARTICLE INFO

*Keywords:*
Nickel thin sheets
Differential speed rolling
EBSD
Texture
Mechanical properties

ABSTRACT

Results of the first study on a microstructure, texture and resultant mechanical properties evolution of technically pure nickel thin sheets (with a thickness of 300 μm) fabricated by differential speed rolling, are shown in the present paper. It was documented that the introduction of a high ratio rolls speed mismatch *R* to the cold rolling process results in a very prominent increase of tensile strength of the material. The recorded ultimate tensile strength of the material cold deformed with the highest $R = 4$ was over 30% higher than that of its counterpart subjected to the conventional (equal speed) rolling, despite the same applied thickness reduction in both cases. Moreover, a comparison with reported literature data shows that DSRed nickel sheets exhibit a comparable or even better strength than their counterparts processed by competitive fabrication methods.

## 1. Introduction

Structural applications of pure metals are generally strongly limited by their insufficient mechanical strength. Despite of having excellent thermo physical properties, a low load bearing ability of pure metals commonly exclude them from a practical engineering usage. A good example of such a case is pure nickel. This material in the form of thin sheets/foils has been recognized as attractive for many large-scale industrial applications, e.g. in nuclear steam generators [1], heat exchangers employed in the soda industry [2] or in micro electro mechanical system (MEMS) [3]. A very good formability and high resistance to stress corrosion cracking under pressure conditions make nickel foils and sheets reasonable candidates for different kinds of micro-gears, micro-switches, chemical activity micro-sensors, or honeycomb catalysts [4]. However, the relatively poor mechanical strength of pure nickel (tensile yield strength (TYS) of 60 MPa and ultimate tensile strength (UTS) of 320 MPa) [5] must be improved in order to ensure a high reliability of this material in terms of its wide industrial applications. In this regard, some processing routines have been proposed to provide an enhanced strength of pure nickel foils, mostly by refining their grain size. This approach, that is based on a well-known Hall-Petch relationship, has mainly involved the use of electrodeposition (ED) techniques (e.g. the lithography, electroplating, and molding - LIGA process) [6–13] or severe plastic deformation (SPD) processing [14–19]. According to reported experimental results reviewed by Yang et al. [6] the UTS of the LIGA nanocrystalline nickel foils (thickness up to 300 μm) is significantly improved as compared to coarse grained counterparts and it is within the range of 490 to 590 MPa. However, there are several drawbacks of the electrodeposition processing (namely, high costs and time-consumption). Furthermore, this process is also quite difficult to control with regards to foil thickness and uniformity.

In terms of the fabrication of high strength thin sheets, the most industrially preferable techniques are those based on continuous plastic deformation processes that may be easily (and inexpensively) adopted to industrial conditions (e.g. accumulative roll bonding (ARB) or constrained groove pressing (CGP)). Zhang et al. [15] documented that imposition of 8 cycles of ARB to commercially pure nickel results in an enhanced structure refinement that is attributed to the geometric accumulation of shear-strain influenced volumes. Consequently, the ARB processed nickel sheets underwent grain boundary and dislocation boundary strengthening which resulted in a UTS of 820 MPa. Nevertheless, a likely degradation of ARB fabricated sheets due to imperfect layer bonding combined with the multistep nature of the process and a need for careful surface preparation [19] make it less attractive. On the other hand, the CGP process allows for the successful fabrication of nickel sheets with a UTS of 505–640 MPa [16–18]. However, it was reported by Satheesh Kumar and Raghu [17] that the CGP processed nickel sheets suffers from a rough surface finish due to the impression created by the sharp profiles of the grooved dies. Consequently, micro cracks are formed on the sheet surface and require its elimination via a further processing.

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http://dx.doi.org/10.1016/j.matchar.2017.06.006
Received 8 May 2017; Received in revised form 4 June 2017; Accepted 4 June 2017
Available online 05 June 2017
1044-5803/ 

In recent years a differential speed rolling (DSR) technique has been developed as a new processing method for fabrication of high strength metallic sheet materials [20]. The DSR principles may be shortly described as a simple modification of the conventional rolling process by the use of non-equal upper and lower rolls speed. Therefore, the simplicity of tool design, the wide availability of devices, and the continuous nature of the process are prominent advantages of this method. A differentiation of rolls speed results alters the strain state imposed to the rolled material (i.e. introduces a strong through thickness shear strain) that strongly affects a microstructure, a crystallographic texture and related mechanical properties of fabricated sheets. It has been already experimentally confirmed for many metallic engineering materials that the implementation of shear strain (the magnitude of which is controlled by applied rolling reductions and rolls speed mismatch values) allows fabrication of metallic sheets with high quality surfaces [21], uniform ultra-fine grained structures and highly improved mechanical characteristics. The DSR process has been applied to many materials systems including e.g. copper [22,23], aluminum and its alloys [24,25], low carbon steels [26–28], titanium [29–31], magnesium alloys [32,33] and even hardly deformable intermetallic based alloys [34–36]. According to Ko et al. [37], on the basis of obtained structural features and mechanical properties, the DSR method should be considered as one of the most effective SPD technique. It has been documented that strength properties of aforementioned DSRed materials are in most cases substantially better than that of their counterparts processed by the conventional (equal) speed rolling and are also competitive to those exhibited by materials subjected to less effective SPD methods.

However, so far there is a lack of available reported data concerning the possible usage of DSR method to a fabrication of high strength nickel foils. Therefore, in this paper we show for the first time the effect of DSR processing on microstructure, texture, and mechanical properties evolution in commercially pure nickel foils.

## 2. Materials and Methods

### 2.1. Materials Processing

The material investigated was commercially pure nickel (99.95 %) in the form of plates. The content of trace elements declared by the producer (STANCHEM, Poland) is as follows: 0.0002 C, 0.0001 Cu, 0.0001 Fe, 0.0002 Pb, 0.0002 S, 0.0002 Zn (in weight %). The material in the as-received condition was subjected to a preliminary thermomechanical processing (including cold rolling with intermediate anneals) in order to obtain initially fully recrystallized structure ensuring a high susceptibility to plastic deformation. After that, nickel plates were electrodischarge machined to obtain bars with dimensions of 100 mm × 25 mm × 1 mm (length × width × thickness) for further experiments.

The main plastic deformation processing was carried out by using a sexton-type rolling mill equipped with working rolls made of tungsten carbide and having equal diameter of 85 mm. The applied processing included a 2-pass cold rolling under dry conditions to a thickness reduction of 70% (i.e. the final thickness of the samples was ~300 μm). The peripheral speed of upper roll was constant (2 m/min), while that of the lower one was adjusted in order to maintain the following rolls speed mismatch $R$ during the experiments:

- $R = 1$ – equal speed rolling (as a reference method);
- $R = 2$, 3 and 4 – differential speed rolling (DSR), where the peripheral speed of upper roll was 2-, 3-, 4-times smaller than that of the lower one, respectively.

By taking into account the documented [38] beneficial impact of strain path alteration on a more homogeneous strain distribution and microstructure evolution in a DSR processed material, the sample orientation was changed "back and forth" (i.e. the rolling direction was reversed) between the consecutive rolling passes. More detailed description of the deformation process is shown elsewhere [34].

### 2.2. Materials Characterization

Microstructural and microtextural features of nickel plates in as-annealed and cold deformed conditions were evaluated by using FEI Quanta 3D field emission gun scanning electron microscope (FEG SEM) coupled with TSL automatic electron backscatter diffraction system (EBSD). The analyses were performed on mounted longitudinal sections of samples. The surface preparation was conducted by means of a mechanical grinding with 250–4000 SiC papers followed by a mechanical polishing with 3–0.25 μm diamond suspensions; and then a final polishing with 1–0.05 μm silica slurries. For each sample several EBSD scans were performed on mid-thickness areas (with dimensions of ~150 μm × 150 μm), using a step size of 0.5 μm. The raw diffraction data were subjected to a post-processing "cleaning" routine in order to remove "noise" effect from the as-received scans. In this regard, points with neighbor to neighbor confidence index (CI) lower than 0.2 were excluded from the analysis [39]. The crystallographic texture was analyzed by computing orientation distribution functions (ODF) from acquired EBSD data by using Harmonic Series Expansion method and under the assumption of orthonormal or triclinic sample symmetry for conventionally rolled or DSRed specimens, respectively. At least 2000 grains were used to calculate ODFs for each sample. In order to in order to keep consistency with literature data [40] reported for fcc metals subjected to differential speed rolling the microtexture was presented in $\varphi_2 = 0$ and 45° sections of reduced Euler angles space ($\varphi_1 = 0$–90°, $\Phi = 0$–90°, $\varphi_2 = 0$–90°).

The mechanical properties of the samples were determined using Vickers microhardness measurements and in static tensile tests. For each sample the microhardness measurements (using a 200 g load and 10 s dwell time) were conducted at 10 randomly selected sites of longitudinal section and then average values were calculated. The static tensile tests were conducted at room temperature, with a strain rate of $1.33 \times 10^{-3}\,s^{-1}$ by using Instron 8501 testing machine. Dog bone shaped samples for tensile tests with a gauge length of 12.5 mm were machined via wire EDM device with the stress axis parallel to the rolling direction. Three tensile specimens of each material condition were tested.

## 3. Results and Discussion

### 3.1. EBSD Microstructure Evaluation

Fig. 1 presents a set of EBSD unique grain color maps taken from nickel samples in initial state and after the various cold rolling processes.

It is found that cold rolling to a 70% thickness reduction transforms the initially equiaxed grain structure (with a mean grain size of ~35 μm) (Fig. 1a) into one composed of grains highly elongated into the rolling direction. However, a comparison of data obtained for the sample cold rolled with equal speed of both rolls ($R = 1$, Fig. 1b) with that of the DSRed samples (Fig. 1c–e), shows that the introduction of rolls speed differentiation leads to the presence of fine grains preferentially located along primary grain boundaries or deformation bands. Furthermore, the quantitative analysis of these maps revealed that the fraction of newly formed fine grains (with a size up to 4 μm) increases with rising the rolls speed mismatch (reaching ~2, ~9, ~11 and ~15% for the $R = 1$, 2, 3 and 4 samples, respectively). These findings clearly point toward a possible activation of structure restoration phenomena (namely, a dynamic or post-dynamic recovery and recrystallization) due to using non-equal rolls speeds. In order to show the through-thickness microstructure homogeneity of the Ni sheets lower magnification inverse pole figure maps for the $R = 1$ and