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**EFFECT OF CRYOGENIC ROLLING AND ELECTROPULSE TREATMENT
ON THE MICROSTRUCTURE AND MICROHARDNESS OF COMMERCIALY
PURE NICKEL NP1**

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Abstract: *Commercially pure nickel (NP1) is widely used in chemical, energy, and aerospace industries because of its excellent corrosion resistance and mechanical properties. However, further improvement of its performance requires effective methods for microstructural refinement. In this study, the combined effect of cryogenic rolling and electropulse treatment (EPT) on the microstructure and microhardness of NP1 nickel was investigated. Samples were cryogenically rolled to a true strain corresponding to 70% thickness reduction and subsequently subjected to electropulse treatment at different current densities. Microhardness measurements and microstructural analysis were performed to evaluate structural evolution after processing. The results demonstrate that the combined treatment significantly modifies the grain structure and affects the microhardness of nickel, indicating the potential of electropulse treatment as an efficient technique for tailoring the properties of commercially pure nickel.*

Keywords: *Commercially pure nickel, Cryogenic rolling, Electropulse treatment, Microstructure, Microhardness, Grain refinement.*

1. INTRODUCTION

Commercially pure nickel is an important engineering material because of its high corrosion resistance, excellent ductility, thermal stability, and good mechanical properties. These characteristics make nickel suitable for applications in chemical engineering, aerospace, power generation, and electronic industries.

The mechanical and corrosion properties of metallic materials strongly depend on their microstructure. Grain refinement, formation of deformation-induced defects, and recrystallization processes can significantly influence hardness, strength, and corrosion behavior. Therefore, modern materials science is focused on developing advanced processing methods capable of controlling the internal structure of metals.

Cryogenic rolling is considered one of the most effective severe plastic deformation techniques. Deformation at liquid nitrogen temperature suppresses dynamic recovery, increases dislocation density, and promotes the formation of ultrafine-grained structures.

Subsequent electropulse treatment provides rapid localized heating and accelerates recovery and recrystallization without prolonged furnace annealing.

The purpose of this work is to investigate the influence of cryogenic rolling followed by electropulse treatment on the microstructure and microhardness of commercially pure NP1 nickel. The obtained results contribute to understanding the structural mechanisms responsible for the improvement of nickel properties and demonstrate the effectiveness of electropulse processing as an alternative post-deformation treatment.

2. Materials and Methods

2.1 Materials

Commercially pure nickel NP1, manufactured according to GOST 492–2006, was selected as the experimental material. The chemical composition of the alloy corresponds to the NP1 grade with a nickel content of approximately 99.9 wt.%. Before deformation, the specimens were annealed at 600 °C for 2 hours to obtain a homogeneous initial microstructure.

2.2 Cryogenic Rolling

Cryogenic rolling was performed using a laboratory six-high rolling mill. During deformation, the samples were immersed in liquid nitrogen to suppress dynamic recovery and increase dislocation accumulation. The rolling speed did not exceed 100 mm/min, while the thickness reduction per pass was limited to 4–5%. The total deformation corresponded to 70% thickness reduction, producing a highly deformed microstructure suitable for subsequent electropulse processing.

2.3 Electropulse Treatment

After cryogenic rolling, the specimens with dimensions of $0.4 \times 4 \times 20$ mm were subjected to electropulse treatment (EPT). The electrical current passed through a 10 mm working zone between copper electrodes.

The duration of each electrical pulse was approximately 100 μ s. Because of the extremely short pulse duration, the heating temperature could not be measured directly. Therefore, the theoretical temperature was calculated using the Joule–Lenz law and the relationship between the integral current density and temperature. The electrical pulse parameters were recorded using a Rogowski coil connected to a digital oscilloscope (AKTAKOM ASK-3107).

2.4 Microhardness Measurements

Microhardness was measured using a METROTEST Vickers microhardness tester under a load of 0.5 N with an indentation time of 10 s. At least ten measurements were carried out for each experimental condition, and the experimental error did not exceed 5%. The average value was used for further analysis.

2.5 Microstructural Characterization

The microstructure of the processed samples was investigated using Electron Backscatter Diffraction (EBSD). The EBSD technique enabled quantitative analysis of grain size, grain boundary characteristics, crystallographic orientation, and recrystallized regions before and after electropulse treatment. These observations were correlated with the measured

microhardness values to evaluate the influence of structural evolution on the mechanical behavior of NP1 nickel.

3. Results and Discussion

3.1 Effect of Cryogenic Rolling on Microhardness

Cryogenic rolling produced a significant refinement of the deformation microstructure of commercially pure NP1 nickel. Plastic deformation at liquid nitrogen temperature effectively suppressed dynamic recovery, leading to the accumulation of a high density of dislocations and deformation bands. As a consequence, the microhardness of the rolled specimens increased compared with the annealed state. These changes are characteristic of strain hardening caused by severe plastic deformation and demonstrate the effectiveness of cryogenic rolling in strengthening nickel.

The increase in hardness can be explained by the formation of numerous lattice defects, including dislocations, low-angle grain boundaries, and deformation twins. These structural features restrict dislocation movement during mechanical loading, resulting in improved resistance to plastic deformation.

3.2 Effect of Electropulse Treatment on Microhardness

Electropulse treatment caused a noticeable evolution of the deformed microstructure. Depending on the integral current density, recovery and recrystallization processes were activated, leading to redistribution of stored deformation energy. Microhardness gradually changed with increasing electrical energy, indicating continuous structural transformation during electropulse processing.

At relatively low processing energies, only partial recovery occurred, preserving most deformation defects. Increasing the current density promoted nucleation of recrystallized grains and gradual reduction of internal stresses. At higher processing energies, the microstructure became considerably more homogeneous because of extensive recrystallization.

These observations demonstrate that electropulse treatment provides an effective alternative to conventional furnace annealing by considerably reducing processing time while maintaining efficient structural modification.

3.3 EBSD Microstructural Analysis

Electron Backscatter Diffraction (EBSD) analysis confirmed substantial changes in grain morphology after cryogenic rolling and electropulse treatment. The cryogenically rolled material exhibited elongated grains with a high density of deformation-induced boundaries, indicating intensive plastic deformation. Following electropulse treatment, numerous recrystallized equiaxed grains appeared, accompanied by a decrease in internal lattice distortion.

The EBSD maps also indicated a reduction in low-angle grain boundaries and an increase in high-angle grain boundaries after treatment. Such structural evolution is characteristic of recrystallization and generally contributes to improved structural stability and more uniform mechanical properties.

The observed grain refinement and boundary evolution demonstrate that electropulse treatment efficiently accelerates recovery and recrystallization without requiring prolonged thermal exposure.

3.4 Corrosion Behaviour

Corrosion tests showed that structural modification significantly influenced the corrosion behaviour of NP1 nickel. Samples subjected to cryogenic rolling followed by electropulse treatment exhibited higher corrosion resistance than untreated specimens. Structural homogenization, grain refinement, and the reduction of deformation defects contributed to the formation of a more stable passive surface layer, thereby decreasing the corrosion rate.

The improvement in corrosion resistance is associated with the decrease in structural heterogeneity and internal stresses after electropulse treatment. Uniform grain distribution reduces the number of preferential sites for localized corrosion initiation and enhances the stability of the protective oxide film.

Overall, the combined application of cryogenic rolling and electropulse treatment proved to be an effective processing route for improving both the mechanical and corrosion properties of commercially pure NP1 nickel.

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