HIGH PRESSURE TORSION OF NICKEL POWDERS OBTAINED BY ELECTRODEPOSITION

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ABSTRACT

A new synthesis route for the production of bulk nanostructured materials is presented. Fine Ni powder was made by selected appropriate electrolysis conditions. A compact material with an average grain size below 40 nm was obtained by subsequent cold pressing. Then, using the high pressure torsion (HPT) deformation technique dense bulk nanocrystalline Ni was achieved. The detailed structural investigations of the as-prepared and HPT deformed Ni powder, including X-ray diffraction (XRD) and transmission electron microscopy (TEM), reveal in both cases the presence of a face centered cubic (FCC) phase without presence of any oxides. Coherently scattering domain size measurements by XRD show a value of 24 nm for the as-deposited powder and an even smaller value of 13.5 nm after HPT deformation. In addition, optical emission spectroscopy was employed to determine the impurity content of the obtained nanostructured material, showing a relatively low content of 0.9 % carbon and oxygen. The microhardness increased after deformation from (1.5 ± 0.08) GPa for the as-deposited Ni powder to (6.6 ± 0.2) GPa for the HPT deformed Ni powder.

Key words: electrodeposition, high pressure torsion, powder consolidation, hardness, bulk nanocrystalline Ni.

INTRODUCTION

Bulk nanocrystalline metals containing a large volume of grain boundaries receive increasing interest since they often show improved mechanical properties as high strength combined with high ductility [1]. Bulk metals are frequently made nanocrystalline using a top down approach (e.g. high pressure torsion [2-3]), but the final grain size that can be reached is limited. On the other hand bottom up approaches (e.g. electrodeposition [4]) allow the production of nanocrystalline metals with considerable smaller grain sizes, but the resulting material is present in the form of powders or thin films.

METHODS OF SAMPLE MANUFACTURING AND ANALYSIS

Ni powders were obtained galvanostatically at a selected high constant current density. As-deposited powders, compactified by cold pressing and deformed

by HPT at room temperature under a hydrostatic pressure of 8 GPa (*Fig. 1*) were systematically studied by X-ray diffraction analysis and TEM methods.

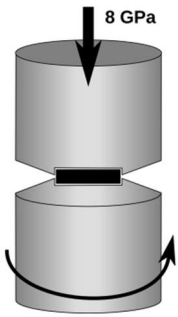

Fig. 1 – Sketch of the HPT deformation technique. The upper and the lower plunger (grey, cylindrical) are pressed against the disk-shaped sample (black). The lower anvil is rotated against the upper one, leading to a torsional deformation in the sample

The impurity content was determined by optical emission spectroscopy.

RESULTS AND DISCUSSION

Figure 2 shows X-ray diffraction patterns of the as-deposited and the HPT deformed Ni powder. Coherently scattering domain size measurements by XRD revealed a value of 24 nm for the as-deposited powder. By the subsequent HPT deformation, the coherently scattering domain size was decreased even further to 13.5 nm.

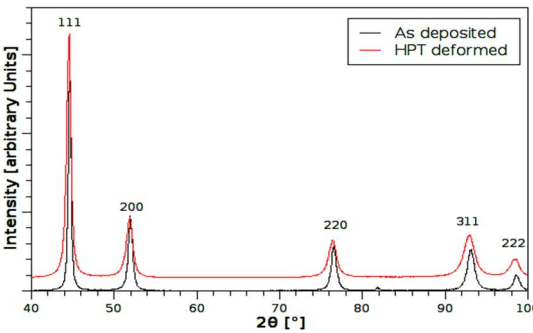

Fig. 2 – X-ray profiles of as-deposited and HPT deformed Ni powder, showing the presence of FCC Ni only.

The low content of impurities and the absence of oxides are crucial for the mechanical properties as impurities would cause embrittlement in the material. Glow discharge optical emission spectroscopy shows a composition of 99.1 at.% of Ni and 0.9 at.% O and C. The TEM study (*Fig. 3*) shows a comparable grain-size of around 35 nm for the as-deposited and HPT deformed Ni powder.

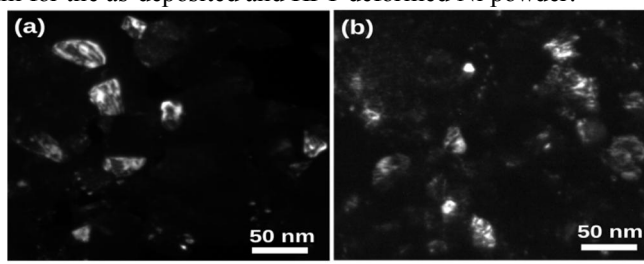

Fig. 3 – Dark field images of (a) as-deposited and (b) HPT deformed Ni powder. The grain size of both samples is comparable (35 nm), but the HPT deformed sample contains a high density of dislocations.

Due to the small grain size a very high microhardness of 6.6 GPa was achieved in the case of the HPT deformed Ni powder.

CONCLUSIONS

We show that by combining electrodeposition and HPT deformation it is possible to obtain dense bulk specimens with small grain sizes. Preliminary results obtained by XRD and TEM analysis show that the very fine grain size of the as-deposited powder (35 nm) is preserved after HPT deformation. The coherently scattering domain size is reduced during the HPT deformation due to the generation of dislocations in the sample. The small grain size and high dislocation density in the achieved specimen leads to an enhanced microhardness. It can be concluded that the combination of electrodeposition and HPT deformation presents a new route for synthesizing bulk nanostructued metals. A dense specimen with a low content of impurities, a very small grain size and improved mechanical properties was achieved.

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