The Effect of Annealing on Hardness in Bulk Nanocrystalline Nickel

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ABSTRACT

Experiments on Hardness were conducted at room temperature on samples of electrodeposited (ED) nanocrystalline (nc) Ni that were annealed at temperatures, ranging from 323 K to 693 K. The results showed that hardness initially increased slightly with increasing annealing temperature and then it decreased rapidly with increasing temperature above 500 K. It was suggested that the increase in hardness below 500 K is due in part to the formation of annealing twins, which serve as a source of strengthening.

INTRODUCTION

Nanocrystalline (nc) materials are defined as single or multiphase polycrystals that are characterized by grain sizes in the range of 1-100 nm. The mechanical properties of nc-materials have been the subject of a number of studies motivated in part by a growing interest in assessing the potential of nc-materials as materials for engineering applications. In particular, the hardness of nc-materials have received considerable attention. Studies on hardness in nc-materials focused in part on the identification of parameters that might influence hardness in terms of softening or strengthening as a result of annealing.

OBJECTIVE

The objective of the present study is to investigate the variations of hardness in nc-Ni prepared via electrodeposition (ED) as a function of annealing temperature.

MATERIALS

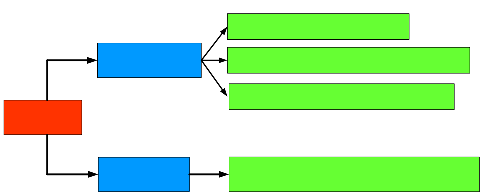
ED nc-Ni used in the present investigation was provided by Integran Technologies Inc. of Toronto, Canada in the form of 0.5 mm sheets with initial average grain size of 20 nm. The ED route of production was used because it can produce a fully dense nc-Ni with a fairly even grain size distribution, and due to the absence of columnar structures within the cross section of the nc-Ni sheets.

Table 1. Chemical composition of electrodeposited nc-Nickel

Elements	C	Si	P	S	Cu	Co	B	Ni
Wt.%	0.013	<0.001	0.003	0.058	0.023	0.071	0.0091	Balance

ED nc-Ni samples were annealed at nine different temperatures ranging from 323 K to 693 K, at two different annealing times, 1 h and 25 h. Samples were mounted and polished to a mirror finish for microstructure analysis and microhardness testing.

EXPERIMENTAL PROCEDURE



RESULTS

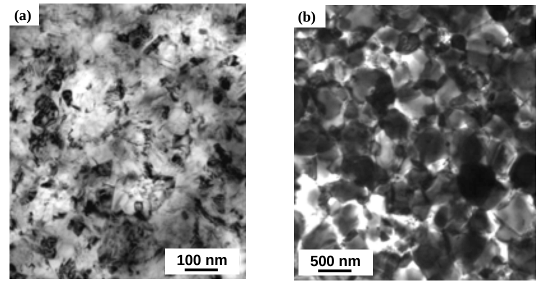


Fig. 1 TEM bright field micrographs of 20 nm nc-Ni annealed at: **(a)** 443 K for 25h and **(b)** 693 K for 1 h.

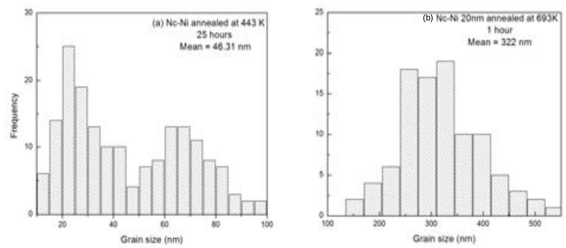


Fig. 2 Grain size distributions determined from TEM of 20nm nc-Ni annealed at: **(a)** 443 K for 25 h and **(b)** 693 K for 1h.

Bimodal grain size distribution with abnormal grain growth was observed for 20 nm nc-Ni samples annealed at 443 K for 25 h. In the temperature range greater than 500 K, significant grain growth occurred and the grain size followed a normal distribution in nc-Ni. An example for the occurrence of significant grain growth was shown in the presence of large grain sizes (>100 nm) in the microstructure of samples annealed at 693 K for 1 h.

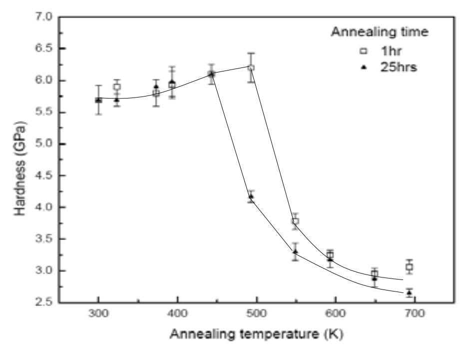


Fig. 5 Room-temperature microhardness of 20nm nc-Ni as a function of annealing temperature.

Hardness was plotted against annealing temperatures for 20 nm nc-Ni samples annealed for two different holding times, 1h and 25 h, in the temperature range 323 – 693 K. Closer inspection of the figure reveals two regions, depending on annealing temperatures. In the low-temperature region (T < 500 K), the hardness increases slightly with increasing grain size, while the holding time has no significant effect on the value of hardness. In the high-temperature region (T > 500 K), hardness decreases with increasing grain size. Also, in this region, hardness varies with the holding time: the higher the holding time, the lower the hardness.

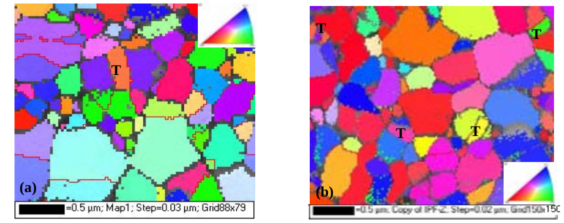


Fig. 3 EBSD orientation map of annealing twins in 20 nm nc-Ni annealed at **(a)**593 K for 1 h and **(b)** 593 K for 25 h.

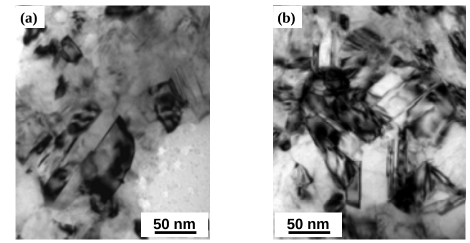


Fig. 4 TEM micrographs of annealing twins in 20 nm nc- Ni annealed at: **(a)** 443 K for 25 h, and **(b)** 493 K for 1 h.

Twins were also observed at temperatures > 500 K during normal grain growth as revealed by the EBSD orientation map where twins are denoted by "T". The density of twins was lower in samples annealed at temperatures < 500 K. An examination of TEM micrographs also indicated the presence of twins in the large grains of the samples that were annealed at 443 K for 25 h and 493 K for 1 h. These twins originated during the annealing process, when deformation was not involved in the experimental procedure (where only heat treatment was carried out). Thus, the induced twins were referred to as annealing twins.

CONCLUSIONS

As a result of this investigation, the following observations can be made:

- Variations in hardness of ED nc-Ni with annealing temperature define two regions: a low temperature region (T < 500K) and a high temperature region (T > 500K).
- Increase of hardness in ED nc-Ni in the low temperature region can be attributed to the presence of annealing twins and the relaxation at non-equilibrium grain boundaries.
- Softening in ED nc-Ni in the high temperature region is attributed to the occurrence of significant grain growth.

REFERENCES

[1] ASTM Standards E-92 and E-384

ACKNOWLEDGMENTS

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