

# EFFECTS OF HEAT TREATMENT ON THE CREEP BEHAVIOR OF ELECTRODEPOSITED NANOCRYSTALLINE NICKEL

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### Abstract

Nanocrystalline (nc) materials have grain sizes in the range 1-100 nm and possess high potential in engineering applications due to their enhanced mechanical properties. The present investigation studies how heat treatment affects the creep behavior of nc-Nickel with an average grain size of 20 nm. The nc-Nickel was produced via an electrodeposition (ED) process and tested using a double shear creep testing technique. Results show that varying the time of heat treatment lowers the steady-state creep rate, effectively enhancing the creep resistance of nc-Ni. Furthermore, an examination of the microstructure of nc-Ni at Transmission Electron Microscopy (TEIM) shows grain growth among the heat treated samples. The presence of annealing twins is also shown in micrographs and reveals its role in increasing the material's resistance to deformation.

#### Introduction

Creep is the time-dependent deformation of a material subjected to constant load or stress. Although the study of creep in nc-materials has been conducted previously, the possibility of inhibiting creep via the use of nanocrystalline materials has yet to be fully understood. For metals, creep is an important factor to consider at elevated temperatures, specifically at or above 0.4 of the material's melting temperature [1]. Because the deformation is permanent, creep is an important design parameter that must be accounted for in engineering applications that operate at high temperatures and are subjected to small loads.

#### Objective

The primary purpose of this study is to investigate the effects of heat treatment on the creep behavior of nc-Nickel.

#### 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 fully dense and pure nc-Ni with a fairly even grain size distribution [2,3]. Double sheer tests have proven to be advantageous in previous creep studies [4,5].

#### Experiment



Figure 1—(a) Double shear creep testing apparatus. (b) Experimental procedure. (c) Double shear test specimen schematic.



Figure 2 shows creep curves for As-received and nc-Ni annealed for various times at 170°C. Creep strain is plotted against the elapsed time of each test, resulting in creep curves that show the presence of a well defined secondary, steady-state creep region that forms approximately after 20 hours.

Strain Rate vs. Annealing Time				Table 1		
L4x10 <sup>7</sup> L2x10 <sup>7</sup> L0x10 <sup>7</sup> U0x10 <sup>4</sup> S0x10 <sup>4</sup> U1x10 <sup>4</sup> L0x10 <sup>4</sup> S0x10 <sup>4</sup> U1x10 <sup>4</sup> 20x10 <sup>4</sup>	Figure 3	0.10 0.09 0.08 0.07 0.05 000 0.05 000 0.03 0.02		Annealing Time (hr)	Strain Rate (1/s)	Offset
				0.00	1.11x10 <sup>-07</sup>	0.0961
				1.00	5.86 x10 <sup>-08</sup>	0.0843
				2.00	2.75 x10 <sup>-08</sup>	0.0404
				5.00	1.52 x10-08	0.0326
		0.01		8.00	2.64 x10 <sup>-08</sup>	0.0602
	0 2 4 6 8 Annealing Time (hr)	10				

Figure 3 demonstrates decreasing strain rate and strain offset with increasing annealing time. Table 1 gives the values of the steady-state creep rates for each annealing temperature. It is evident that the creep rate decreases with increasing annealing temperature, however the eight hour annealing time deviates from this overall trend.



Table 2 presents the average grain size for the as-received nc-Ni, as well as for the annealed nc-Ni before deformation. Table 2 and Figure 4 show that increasing the annealing time resulted in an increase in the average grain size. Although the specimen annealed for one hour did not have significant grain growth, there is significant grain growth for nc-Ni annealed for two, five, and eight hours. TEM Micrographs and Grain Size Distributions





Figure 5 shows TEM micrographs of nc-Ni (a) AR and annealed at  $170^{\circ}$  C for (b) one hour, (c) two hours, (d) five hours, and (e) eight hours. An examination of the annealed samples micrographs shows the presence of special structures known as annealing twins within some of the grains. An annealing twin results in a symmetrical atomic arrangement within a grain, causing it to divide due to a twin boundary [1]. These boundaries act as a barrier to dislocation movement, adding to dislocation immobility, and further enhancing the material's resistance to deformation.









Figure 6a shows the normal distribution of grain size for as-received nc-Ni. Figures 6b, c, d, and e show the size distributions for nc-Ni annealed at  $170^{\circ}$  C for one, two, five and eight hours, respectively. Figures 6b-e show that significant grain growth has occurred. These histograms depict two peaks in their distributions. The first peak represents approximately the average grain size of the as-received nc-Ni, the second shows a high number of larger grain sizes. This indicates that during the annealing process, abnormal grain growth occurred.

#### Conclusions

 Heat treatment lowered the creep strain and steady-state creep rate for nc-Ni annealed at 170°C for times of one, two, five, and eight hours. It can be concluded that heat treatment increases the creep resistance for electrodeposited nc-Ni at this temperature range.

- 2. Annealing the nc-Ni resulted in grain growth for the annealed specimens.
- 3. The presence of annealing twins within the microstructure of the annealed nc-Ni gives an insight as to how the material was able to increase its resistance to deformation despite an increase in its average grain size. Twinning boundaries enhanced dislocation immobilization, thus giving the material more resistance to mechanical deformation.
- 4. Although less than the creep strain of AR nc-Ni, the eight hour heat treated nc-Ni did not perform as well as the nc-Ni annealed for a less
- amounts of time. A five hour annealing time proved to have the highest creep resistance, thus making it the optimal time for heat treatment.

## References

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