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Mechanical spectroscopy of nanocrystalline nickel near room temperature

J. Lohmiller,[a,b] C. Eberl,[b] R. Schwaiger,[b,c] O. Kraft[b,c] and T.J. Balk[a,*]

[a] *Chemical and Materials Engineering, University of Kentucky, 177 F. Paul Anderson Tower, Lexington, KY 40506, USA*
[b] *Universität Karlsruhe (TH), Institut für Zuverlässigkeit von Bauteilen und Systemen, Kaiserstr. 12, 76131 Karlsruhe, Germany*
[c] *Forschungszentrum Karlsruhe, Institut für Materialforschung II, Postfach 3640, 76021 Karlsruhe, Germany*

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In this study, the anelastic behavior of nanocrystalline nickel was investigated by mechanical spectroscopy. Test temperature ranged from room temperature to 415 K, and frequency from 235 to 2567 Hz, with two internal friction peaks observed. Peak temperature increased with increasing frequency, indicating a thermally activated damping process. The smaller peak yields an activation energy $H = 0.70$ eV. The peaks appear to be P_β and P_γ Hasiguti peaks, arising from damping due to dislocations and point defects.

Keywords: Anelasticity; Damping; Internal friction; Nanocrystalline nickel

Anelasticity studies have a long tradition in physical metallurgy, starting with the fundamental work of Zener [1] in the 1940s. Anelasticity describes the time-dependent elastic behavior of a material, i.e. when stress is applied and a phase lag δ is observed between stress and resultant strain. This behavior occurs during low-amplitude, dynamic loading.

Bonetti et al. [2] studied the damping behavior of nanocrystalline nickel (nc–Ni). However, their observations were obtained at noticeably higher temperatures (up to 1300 K) and their samples (made from ball-milled powders) were different than in the current study. We are not aware of any group that has investigated the damping behavior of nc–Ni over the temperature range covered in our study (room temperature to 415 K). This is, however, an important temperature interval, as the grain size and structure of nc-Ni remain stable.

Several studies near room temperature have been performed on coarse-grained nickel (cg–Ni). Hasiguti et al. [3] observed peaks above and below room temperature for cg–Ni. There was no consensus on the operative mechanism for these so-called Hasiguti peaks, although they appeared to arise from the interaction of dislocations and point defects. Sommer and Beshers [4] reported two peaks for cg–Ni at low temperatures (77–350 K) and high frequency (30 kHz). They observed a large peak P_x at $T_p = 248 \pm 40$ K, which they interpreted as a Bordoni peak. Additionally, they observed a smaller peak P_y on the left shoulder of the Bordoni peak, at $T_p = 138 \pm 7$ K, which was identified as a Niblett–Wilks (N–W) peak. Bordoni and N–W peaks are generally due solely to dislocations, specifically the formation of double kinks and anti-kinks [5]. Bonetti et al. completed several studies on nc–Ni at higher temperatures [2,6,7], where grain boundary (GB) diffusion was found to be the operative mechanism. These results for Ni are summarized in Table 1.

In the current study, damping measurements on nc–Ni were performed with a home-built system [9]. This system includes a high-vacuum chamber with integrated heating stage, laser Doppler vibrometer and data acquisition system. The damping system, which measures the logarithmic decrement of free decay, was originally used for thin films [9–11], but has also been successfully applied here to foils of bulk nc–Ni.

Samples with free-standing cantilevers were cut from 110 μm thick nc–Ni sheet by wire electrical discharge machining. The nc–Ni (Integran Technologies Inc., Toronto, Canada) was produced by electrodeposition, although fabrication details are proprietary knowledge. Average grain size was ~20 nm. Resonant frequency was varied by changing the paddle size at the end of

* Corresponding author. Tel.: +1 859 257 4582; fax: +1 859 323 1929; e-mail: balk@engr.uky.edu

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Table 1. Summary of activation energies and peak temperatures for internal friction studies of Ni in the literature, together with homologous temperature, sample test frequency, general grain size d_g (coarse or nanocrystalline), peak type and governing mechanism

H (eV)	T_p (K)	T_p/T_m	f (Hz)	d_g	Peak type	Mechanism	Ref.
0.31 ± 0.04	155	0.090	≈1,000	cg	Hasiguti: P_α	Interaction of dislocations and point defects	[3]
0.53 ± 0.05	350	0.203	≈1,000		P_β		
0.72 ± 0.06	397	0.230	≈1,000		P_γ		
0.14	131–145	0.076–0.084	≈30,000	cg	N–W	Dislocations: kink/anti-kink pair nucleation	[4,8]
0.40	208–288	0.120–0.167	≈30,000		Bordoni		
1.1 ± 0.1	≈650	0.376	10–10,000	nc	GB	GB diffusion	[2]
1.25 ± 0.15	475 @ 0.2 Hz	0.275 @ 0.2 Hz	0.01–10	nc	GB	GB diffusion	[6]
1.25 ± 0.15	475–660	0.275–0.382	0.01–10,000	nc	GB	GB diffusion	[7]

Internal friction peaks for cg–Ni have been found in the same temperature range as the current study (300–415 K), but for nc–Ni the lowest reported temperature at which peaks have arisen is 475 K.

the cantilever. Paddle size ranged from 2 mm × 2 mm to 8 mm × 8 mm, resulting in a frequency range of 235–2567 Hz. To estimate the resonant frequency for each paddle geometry before sample fabrication, finite element simulations were performed using Ansys®.

Internal friction was measured as a function of temperature for six nc–Ni samples. The heating rate was 0.5 K min^{-1}, with measurements every 4 K. Inverse quality factor Q^{-1} was plotted vs. temperature T to identify internal friction peaks. Experimental curves contained a strong exponential background damping component inherent to polycrystalline metals (see Eq. (1)), in addition to the Debye peak itself (see Eq. (2)):

$$Q_{bg}^{-1} = A\exp(BT) \tag{1}$$

$$Q_{peak}^{-1} = \Delta\frac{\omega\tau}{1+(\omega\tau)^2} \tag{2}$$

where A and B are fitting parameters, ω is the angular frequency of vibration and τ is the relaxation time.

For the background, an exponential curve was fitted to the experimental data, assuming that only background damping exists at temperatures far from the peak. This background was subtracted from the Q^{-1} plot, yielding the peak damping. Data were plotted with Origin® 7.0 and peak fitting functions were utilized to determine the peak temperature as precisely as possible.

An example of a Q^{-1} vs. T plot is provided in Figure 1, showing both the experimental data and the fitted exponential background damping. Over this temperature range, Q^{-1} doubles from ~0.0030 to 0.0060 (see Fig. 1a). Figure 1b shows the peak damping fitted with a log-normal function. Peak height is ~0.0006 above background and peak temperature is $T_p = 354.6$ K. A complete list of peak temperatures and corresponding room temperature frequencies is provided in Table 2.

To determine if grain growth occurred during thermal cycling, X-ray diffraction was used to measure broadening of the (1 1 1) and (2 0 0) peaks in θ–2θ scans. This straightforward method employs the Scherrer equation and provides accurate results for grain sizes below 50 nm [12]. Instrumental broadening ($\Delta\theta = 0.045°$) was subtracted from the width of nc–Ni peaks before application of the Scherrer equation. No significant microstrain is expected in the nc–Ni samples since these were electrodeposited, and thus any remaining peak broadening should be due to small grain size. No appreciable grain growth was observed in the samples tested here. This is shown in Table 2, which lists grain size data

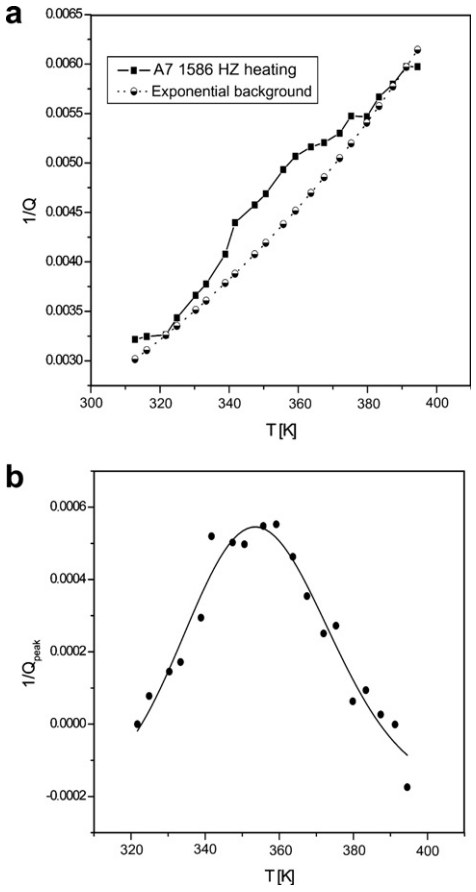

Figure 1. (a) Sample A7 ($f_{0,RT} = 1586$ Hz). A slight but noticeable peak is observed above the background, which is represented by an exponential curve fit. (b) After background subtraction, the plot of inverse quality factor exhibits a clear peak at $T_p = 354.6 \pm 0.8$ K.

before and after thermal cycling. For example, sample A2 had an as-received grain size of 19.5 nm (according to the (1 1 1) peak) or 13.9 nm (according to the (2 0 0) peak). After five thermal cycles and 9 h of cumulative annealing between 370 and 386 K, X-ray measurements indicated a grain size of 20.4 or 14.4 nm, respectively. This deviation is small and within the error of measurement. The finding that grain size determined from the (1 1 1) peak is ~1.5 times larger than that from the (2 0 0) peak agrees with the results of Thuvander [13].

For some samples, another much larger peak also appeared during continued heating, as shown in Figure 2. It was not decisively determined why these larger peaks