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Observation of local internal friction and plasticity onset in nanocrystalline nickel by atomic force acoustic microscopy

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Abstract

Atomic force acoustic microscopy (AFAM) is a near-field microscopy technique exploiting the vibrational behavior of the atomic force microscope cantilever. With its tip in contact with a surface, it is sensitive to its elastic and anelastic properties. We show how AFAM can be used to investigate the onset of plasticity in nanocrystalline nickel. To this end cantilever resonance curves are recorded with varying tip-loading force P. From the resonance frequencies and the width of the resonance curves, one obtains the contact stiffness k^* and the contact damping Q^{-1}. Plotting these quantities vs. P, one observes damping peaks, as well as a reduction of the contact stiffness at specific P ($\approx$1 μN) due to the nucleation of partial dislocation loops at grain boundaries. The local Q^{-1} value is most likely caused both by the nucleation and by the interaction of the loop with the phonon and the electron baths. There is a background damping which is related to the global ultrasonic absorption.

Keywords: Nanocrystalline materials; Anelasticity; Atomic force acoustic microscopy (AFM); Dislocations; Acoustic methods

1. Introduction

Nanocrystalline (nc) materials possess a microstructure with characteristic lengths in the nanometer range. These materials exhibit physical and mechanical properties different from their polycrystalline counterparts and are attributed to their high grain boundary volume [1]. For example, an inverse Hall–Petch relation for grain sizes below 10 nm has been observed [2–5]. This implies that for plastic deformation, mechanisms must operate in addition to dislocation motion known from polycrystalline materials [6]. Computer simulations of nc-materials gave some insights into the dynamics of the microstructure under the action of temperature and/or stress [7,8]. From such investigations the occurrence of several plastic deformation mechanisms as a function of decreasing grain size has been postulated: dislocation movement, grain boundary-sliding, and grain boundary-rotation for the smallest grain sizes [8–11]. The simulation of indentation experiments in nanocrystalline face-centered cubic (fcc) metals was carried out, in order to investigate the dislocation activity and interaction with grain boundaries [12,13]. In the simulations the indenter sizes are between 1 and 10 nm, which is much smaller than the indenters employed in the experiments, which are several hundred nm. The data obtained from indentation experiments are averages over several grains.

In this work we nucleate dislocations in grains of nc-nickel of sizes between 14 and 76 nm and we investigate their dynamic behavior using atomic force acoustic microscopy (AFAM) [14]. AFAM is a tool for quantitative elasticity measurements [15], providing a spatial resolution of about 10 nm. In this work, using the spectroscopic mode of the AFAM technique, the contact stiffness k^* and the contact damping Q^{-1} vs. loading force P have been

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obtained for nc-Ni with different grain sizes, ranging from 14 to 76 nm. We observe a reduction of the contact stiffness at certain loading forces, accompanied by an increase of the contact-resonance damping, which we assign to the nucleation of heterogeneous partial dislocation nucleation at grain boundaries, and to internal friction of the moving dislocation in the elastic oscillatory field of the AFM cantilever-tip. Finally, we compare the background of the Q^{-1} value to the mechanism known for the ultrasonic absorption processes in metals.

2. Atomic force acoustic microscopy technique

As depicted in Fig. 1, in one version of AFAM, one injects longitudinal waves into the sample from its bottom, leading to periodic displacements of its upper surface. The displacements couple via the tip into the AFM cantilever and excite it to bending vibrations. They are detected with the same optical beam-deflection detector as is used for the topography measurements, provided that the bandwidth of the detector system is sufficiently large.

The free bending-resonances of a given cantilever depend on its geometry, its elastic constants, and its density. The resonances have Q-factors of typically 200–800 [16] in ambient air. Bringing the cantilever into contact, a shift of the resonance frequencies to higher values occurs and the Q-factors are reduced by at least a factor of five (see Tables 1 and 2), due to the stiffening and damping effects of the contact. AFAM can be used as an imaging instrument, where the amplitude of vibration is recorded on the sample surface in a raster scan (Fig. 2). In this case the cantilever is excited at a fixed frequency near its resonance. Depending on the local contact stiffness, the resonance frequency shifts and leads to changes in the vibration amplitude at the working frequency. Likewise, one can use the contact-resonance frequency value as imaging quantity [17]. The recording of contact-resonance curves allows the quantitative evaluation of the local elastic and anelastic properties. Here, we discuss the determination of anelastic properties of nc-Ni. The technique can be applied to other materials as well.

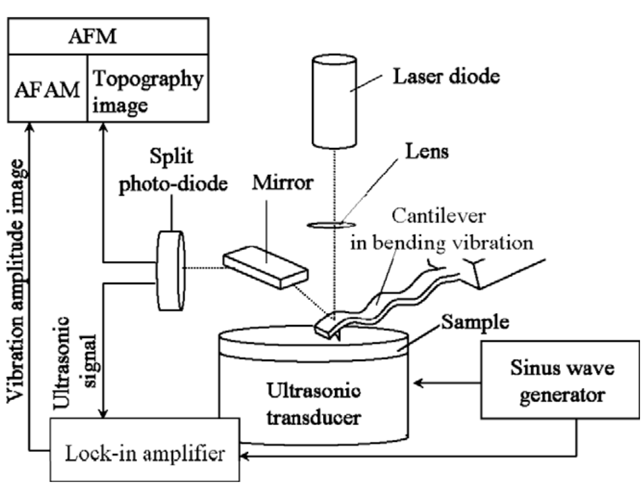

Fig. 1. Experimental set-up of atomic force acoustic microscopy. An ultrasonic transducer excited by a sine-wave generator sets the sample surface into vertical vibrations and via the tip-sample forces the cantilever into bending vibrations. The cantilever vibrations are detected by a beam-deflection detector consisting of a laser diode, a lens, a mirror, and a split photo-diode. The static and low-frequency components of the cantilever deflection are used for the topography image. The high-frequency components are processed by a lock-in amplifier and displayed as an acoustic image.

2.1. 1. Spectroscopic AFAM technique

Oscillating free and clamped elastic beams are treated in many textbooks of mechanical engineering. There is a number of papers describing cantilever oscillations in AFAM experiments [14,15,18,19]. For a forced rectangular beam the following equation of motion holds:

$$EI\frac{\partial^4 y}{\partial x^4} + \eta_{\text{air}}\frac{\partial y}{\partial t} + \rho A\frac{\partial^4 y}{\partial t^4} = F\delta(x - x_0)e^{i\omega t} \tag{1}$$

where E is the Young's modulus of the beam material, I is the cantilever area moment of inertia, η_{air} is the damping constant for frictional losses in air, ρ is the beam mass density, A is its cross-section, and $y(x,\ t)$ is the vertical displacement of the beam. The position along the beam is x and t is the time. F is the force which acts on the tip at the position x_0.

Let us have a closer look at the interactions determining the boundary conditions required to solve Eq. (1). In general, the interactions of the tip with the sample are due to repulsive elastic and attractive adhesive forces. The adhesion forces can be neglected if sufficiently high static loads are applied. For small vibration amplitudes the non-linear interaction forces can be approximated linearly, leading to the mechanical model for the cantilever vibrations shown in Fig. 3a. Often, the contact forces acting on the tip are represented by two linear springs for the vertical and lateral contact stiffness k^* and k^*_{Lat}, and two dash-pots γ^* and γ^*_{Lat} for the vertical and lateral contact damping, respectively, leading to the boundary conditions for the displacement and the slope of the cantilever beam:

$$x_0 = L \text{ or } x' = L' : \begin{cases} y(x) = y'(x') \\ \dfrac{\partial y(x)}{\partial x} = -\dfrac{\partial y'(x')}{\partial x'} \end{cases} \tag{2}$$

A further boundary condition is obtained from the equilibrium between the shear forces arising from the cantilever deflection and the contact restoring forces. In the case where only vertical forces are present, i.e. $\varphi = 0$ and $L' = 0$, one obtains the following equation [18,19]:

$$EI\frac{\partial^3 y(x)}{\partial x^3}\bigg|_{x=L} = k^* y(L,t) + \gamma^*\frac{\partial y(L,t)}{\partial t} \tag{3}$$

The linear response of the cantilever to the ultrasonic excitation are eigenmodes, which are determined by the cantilever stiffness k, the contact stiffness k^*, and the