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# The Nano-Jackhammer effect in probing near-surface mechanical properties

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### ABSTRACT

Because of its ease of implementation and insensitivity to indenter drift, dynamic indentation techniques have been frequently used to measure mechanical properties of bulk and thin film materials as a function of indenter displacement. However, the actual effect of the oscillating tip on the material response has not been examined. Recently, it has been shown that the oscillation used with dynamic indentation techniques alters the measured hardness value of ductile metallic materials, especially at depths less than 200 nm. The alteration in the hardness is due to the added energy associated with the oscillation which assists dislocation nucleation. Atomistic simulations on nickel thin films agree with experiments that more dislocations are nucleated during dynamic indents than with quasi-static indents. Through the analysis of quasi-static and dynamic indents made into nickel single crystals and thin films, a theory to describe this phenomenon is presented. This is coined the Nano-Jackhammer effect, a combination of dislocation nucleation and strain rate sensitivity caused by indentation with a superimposed dynamic oscillation.

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## 1. Introduction

The importance of understanding near-surface mechanical behavior cannot be overstated given the rapidity with which device scaling has occurred (Cheng et al., 2006; Cordill, 2007; Cordill et al., 2009; Cross et al., 2006; Fischer-Cripps, 2004; Mook et al., 2008; Schuh et al., 2005; White et al., 2005). Additionally, the difficulty in atomistic simulations of such properties with realistic temperature and temporal variables has placed a burden of proof on experimental confirmation (Cordill et al., 2009; Cross et al., 2006; Schuh et al., 2005). Two of the techniques of choice have been atomic force microscopy (AFM) and nanoindentation which can drive sharp tips into the near-surface to extract modulus, hardness, flow stress, and creep data, among other mechanical properties. Extracting data with near-surface probes has been made simple at intermediate penetration depths (>100 nm) but perhaps more complicated at very shallow depths (1–50 nm) by incorporating a superimposed oscillatory displacement. Such types of experiments have their origin in internal friction studies (Sklad et al., 1973) and dynamic mechanical property measurement of polymers (Cheng et al., 2006; Fischer-Cripps, 2004; White et al., 2005). It is well known that dislocations respond differently to small strains of different magnitudes at high frequencies (Cheng et al., 2006). Nanoindentation involves much larger displacements (strains) at much lower frequencies where the goal is to understand the response of metallic and ionically bonded materials to frequency modulated oscillations.

Dynamic nanoindentation, also called the Continuous Stiffness Method (CSM), has experienced a large user base since it is simpler to extract modulus and hardness data continuously as a function of depth. This technique allows for the measurement of mechanical properties with one indent since the stiffness is continuously measured. Often with these measurements an indentation size effect arises where the properties near the surface are much higher than the bulk. A microstructurally-based strain gradient plasticity model, first introduced by Nix and Gao (1998) is commonly used to explain the effect. Strain gradient plasticity has been applied by several others to help understand microbend tests (Shi et al., 2008), plasticity (Abu-Al-Rub, 2008; Abu-Al-Rub and Voyiadjis, 2006; Qu et al., 2006; Volokh and Trapper, 2007; Wang et al., 2007), hardening effects (Abu-Al-Rub, 2008; Zhang et al., 2007), interface fracture and plasticity (Abu-Al-Rub, 2008; Siddiq et al., 2007) and the Taylor dislocation model (Hwang et al., 2004; Liu et al., 2005). The main concern is that the dissipative energy associated with even small oscillatory displacements of sharp tips could result in different properties (Cordill, 2007; Cordill et al., 2008), particularly with regard to dislocation nucleation and theoretical shear stress determinations. For example, at the macroscale, it has been known for some time that relatively small resonant-induced displacements on aircraft structures can lead to premature failure. One can lower this to the next familiar scale by envisioning a vibratory etching tool with a 1 mm sharp probe oscillating at microns of displacement. In this case the damage is plastic deformation of the metal surface. With the nanoindenter or AFM, another lowering of three orders of magnitude scale, suggests that micron and less sharp probes oscillating at nanometer displacements must have some similar damage or dissipative effect. It is known that the first yield point is never attained in repetition, even with identical loading conditions. Rather the first yield point scatters statistically (Ngan et al., 2006a; Schuh and Lund, 2004). However, when the statistics are considered (Cordill et al., 2008) the softening phenomena is still observed between quasi-static and dynamic indentation. This is seen in Fig. 1 for a nickel single crystal and a nanocrystalline nickel thin film. The data shown is from 10 indents and averaged as shown by the error bars denoting the standard deviation. There is a clear difference in the first yield point for both testing conditions and for both samples. All quasi-static experiments were performed with the same conditions as well as the dynamic experiments, as discussed later. It will be demonstrated that such nanometer displacements are indeed important in the early stages of deformation associated with contact mechanics. A sampling of some extensive data sets on nickel films (Cordill, 2007) and single crystals (Mook et al., 2008) will demonstrate what is coined here to be the Nano-Jackhammer effect. Just like it is easier to engrave a metal surface with a Micro-Jackhammer without too much external force, it is likewise easier to indent a crystalline surface with a Nano-Jackhammer.

What is proposed is that two strain rate effects may exist simultaneously when oscillatory displacements at different frequencies are superimposed on high mean pressures. The normal strain rate

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