

## Report of Sabbatical Leave Activity, 2005-2006

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### Overview

This document summarizes my activities and major accomplishments during my sabbatical leave for the 2005-06 academic year. Briefly, I spent the sabbatical leave (starting June 2005 at the University of Edinburgh, Scotland) collaborating with members of the Institute of Materials and Processes, and Spring Semester 2006 at Air Force Research Laboratory, Wright-Patterson AFB, Dayton, OH, collaborating with the MLLM Division. The sabbatical leave was very successful in allowing me not only to develop the intended collaborations and to carry out the proposed experiments but also obtain new and exciting scientific results in the emerging area of dislocation micro- and nanoplasticity. As a direct result of our work, one paper has been published in 2005, two have been submitted for publication and three are in preparation. Furthermore, the collaborations fostered during my leave are continuing in the form of joint proposals for future work, which we are writing now.

The major scientific accomplishments of the sabbatical leave can be summarized as follows. Using laboratory and computational facilities at the University of Edinburgh, and in collaboration with Prof. H. Neuhauser, Technical University of Braunschweig, Germany, we studied statistical properties of the surface roughening of solids at nanoscale level as produced by moving dislocations as a function of strain, localized obstacle concentration, and type of solids (pure metals, alloys, ionic crystals). This is a new technique based on new high-resolution instrumentation so that we were able to measure individual nanometer-scale steps left behind moving dislocations during mechanical tests. Hence, the roughness evolution allows us for the first time to identify correlating activity of very large networking dislocation systems in single crystals subjected to large fluctuations. In particular, we demonstrated that such systems exhibit several unusual properties such as long-rang correlation, power law probability distribution, and fractal characteristics. This, in turn, provides insight into the formation of deformation structure and hardening processes in deforming materials, which are both of fundamental and practical interest.

Using well equipped Air Force Research Laboratory in Dayton, OH, we studied size effects in two solids that were investigated in Edinburgh, manufacturing inside an electron microscope micron-scale samples (from 20  $\mu\text{m}$  down to 1 micron) and deforming them in home-made unique high-precision loading system. The crystals under investigation, LiF, were obtained from a crystal growth center in Ohio thanks to a previous collaboration at Edinburgh. The final microsamples, unlike all previously prepared from metals and alloys, had an exceptionally low density of dislocations, and also showed many unusual mechanical properties complementary to those obtained in Edinburgh. Among the most important findings was the direct measurement of the highest yield stress in 1- $\mu\text{m}$  samples not so far from predicted theoretical strength. We demonstrated that the microsamples exhibit very high yield stress independently of their bulk

property so that we observed an increase by a factor of 300 in the yield stress of 1- $\mu\text{m}$  samples compared to the bulk samples.

### **University of Edinburgh, Scotland, June-December 2005**

I spent this time as a Visiting Professor in the School of Engineering and Electronics, Institute for Materials and Processes and Centre for Materials Science and Engineering, at the University of Edinburgh. There I collaborated with Professors M. Zaiser, C. Hall, and V. Koutsos on theoretical, computational, and experimental studies of dislocation induced surface roughness at nanoscales. Our primary effort was to carry out laboratory experiments on large ionic alkali halides (AH) single crystals (LiF, KCl, and NaCl), Cu single crystals, and several polycrystalline alloys. Samples were successively deformed by compression in a test machine starting from the yield stress up to the largest possible deformation. Sample's surface roughness was then measured by Zygo Scanning White Light Interferometer (SWLI) or AFM, both with 1 Å height resolution, after each deformation test (Figure 1).

Using statistical software code developed for such measurements, we were able to obtain unique statistical properties of the deforming samples to characterize the surface morphology evolution, such as 1D surface profiles distributions, components of the plastic strain tensor, global and local surface roughness, height difference correlation functions, and roughness exponents. We demonstrated that during deformation initially almost smooth as-cleaved surfaces developed self-affine roughness over several orders of magnitude in scale. We found a quite unpredicted result that the correlation length was continued to increase with the plastic strain and obeyed the power law dependence. This indicates that all local deformation events such as nucleation of new dislocations and their motion are not completely independent but spatially correlated all over the sample's volume (Figure 2). From roughness exponent we were able to find the Hurst exponent and show that the spatial organization in deforming solids is characterized by random fractal patterns with. The first results of the study have been published in paper "Evolution of self-affine surface roughness in plastically deforming KCl single crystals", E.M. Nadgorny, J. Schwerdtfeger, F. Madani, V. Koutsos, E. C. Aifantis, and M. Zaiser, Proceedings of Science (PoS-SMPRI), 1-11 (2005).

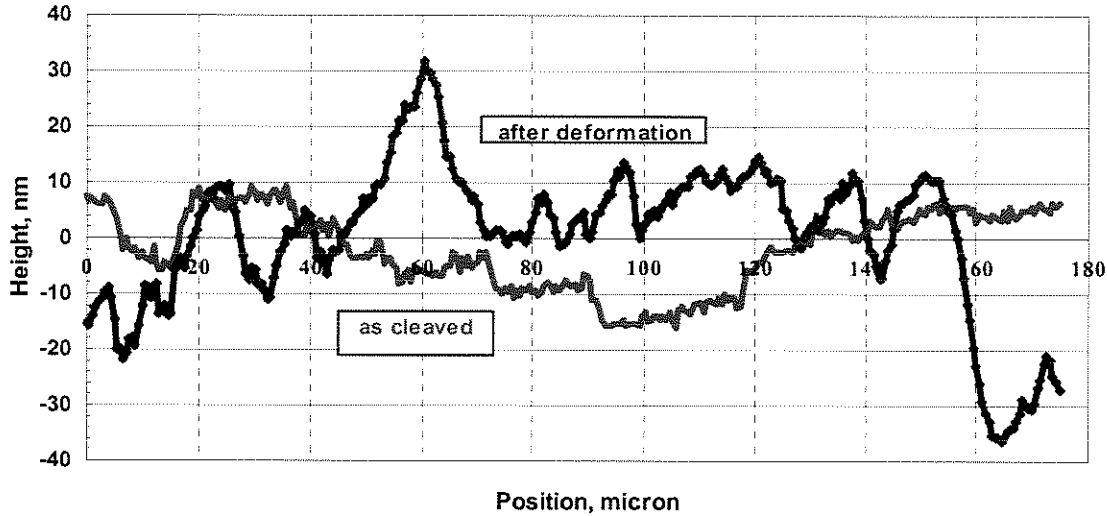


Figure 1. Surface profiles obtained by Zygo SWLI on KCl samples before and after small deformation of  $\sim 0.5\%$ . The height of each dislocation “elementary step”  $h = 0.31 \text{ nm}$ . There are both narrow and wide slip bands appeared after the yield stress.

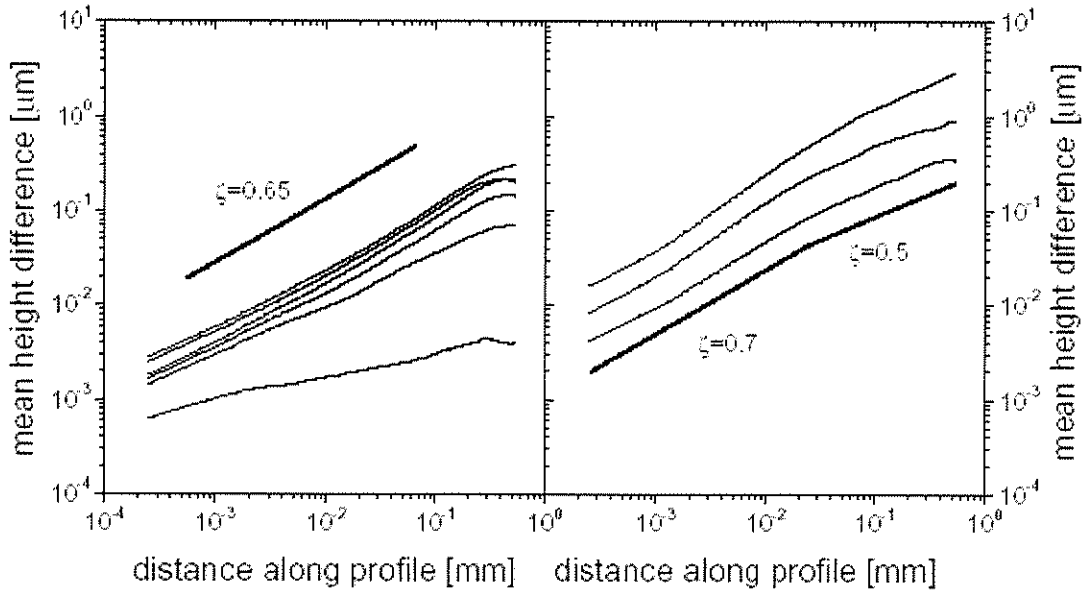


Figure 2. Local roughening of the surface of a KCl single crystal as determined by SWLI (mean height difference vs. distance along profile); left: deformation in Stage I; strains 0%, 0.56%, 1.16%, 1.44%, 2.01% and 2.67%; right: deformation in Stage II; strains 3.45%, 5.00% and 7.12%.

## Air Force Research Laboratory, Dayton, OH, March - June 2006

I spent this time at the AF Research Laboratory, Wright-Patterson AFB, Dayton, OH, where I collaborated with Drs. D. Dimiduk, M. Uchic, and others on a project to study size effects in the same crystalline materials I worked on before in Edinburgh. My collaborators at AFRL recently developed methods for preparing micron-scale samples of different materials via focused ion beam machining and compression testing them in nanoindenter. Their experience allows us to manufacture unique cylindrical LiF samples of diameters 20, 5 and 1  $\mu\text{m}$ , with the ratio length-diameter about 3 (Figure 3). The prepared set of samples was tested on compression at a constant displacement rate of 5 nm/s. Although the deformation processes of LiF are quite different compared to FCC-derivative metals studied before at AFRL, LiF demonstrated similar size-strengthening effects. For example, the measured engineering stress at 1% shear strain in 1- $\mu\text{m}$  LiF samples ranged from 600 to 750 MPa (Figure 4). Such high values are in sharp contrast to the yield stress of as-grown bulk LiF, which is only approximately 2-3 MPa. Applying selective etching technique, we showed that the measured initial dislocation density in all our LiF crystals was very low. After gamma-irradiation, the yield stress of bulk as-grown LiF crystals became almost ten times higher, but such irradiation-hardening left the deformation characteristics of LiF microsamples practically unchanged. For both the as-grown and irradiated LiF micron-scale crystals, the flow stress  $\sigma$  as a function of the sample diameter  $D$  obeys the power law,  $\sigma \sim D^{-\alpha}$ , with  $\alpha \approx 0.8$  (Figure 4). Like metals, LiF crystals also demonstrated the other size-scale effects, such as high micro-plastic hardening rates, transitions to a low work hardening response, and appearance of fast intermittent deformation events or “avalanches” ranging from several nanometers to microns in scale. The obtained results demonstrate that early observations obtained before only on metals are also observed on solids of different type. This confirms the universal nature of the size-scale effects in plasticity, so that existing models should be modified to be in agreement with these new findings. The first results of the study will be presented in two papers at the 2006 MRS Meeting in Boston this November: E.M. Nadgorny, M.D. Uchic, and D.M. Dimiduk, “Size Effects in Lithium Fluoride Micron-Scale Single Crystals”; and D.M. Dimiduk, C. Woodward, M.D. Uchic, S.I. Rao, T.A. Parthasarathy, and E.M. Nadgorny, “Nucleation, Percolation and Size-Scaling in Microcrystal Plasticity”. Two more papers describing in more details these and the other new results, together with first modeling, are in preparation.

During my sabbatical I also visited three Universities, one in Scotland and two in Germany, where I gave seminars on previous and current research. Additionally, I presented two papers at International Meetings in India and Canada. At the International Conference “Statistical Mechanics of Plasticity and Related Instabilities”, Bangalore, India, I presented a paper E. Nadgorny “Dynamics of Discrete Dislocations and Crystal Plasticity”. I also was an Organizer and Chair (together with MTU Adjunct Professor Michael Zaiser) of the Section “Collective Dynamics of Dislocations in Deformed Crystals (Experiments and Modeling): Fluctuations, Instabilities and Size Effects” at the 12th International Symposium, “Plasticity 2006”, Halifax, Canada, where we presented two papers: E. Nadgorny and M. Zaiser, “Experimental Study of Plastic Flow Scale Invariance in Alkali Halides and FCC Metals”, and M. Zaiser and E. Nadgorny, “Theory and Modeling of Scale Invariance of Plastic Flow”.

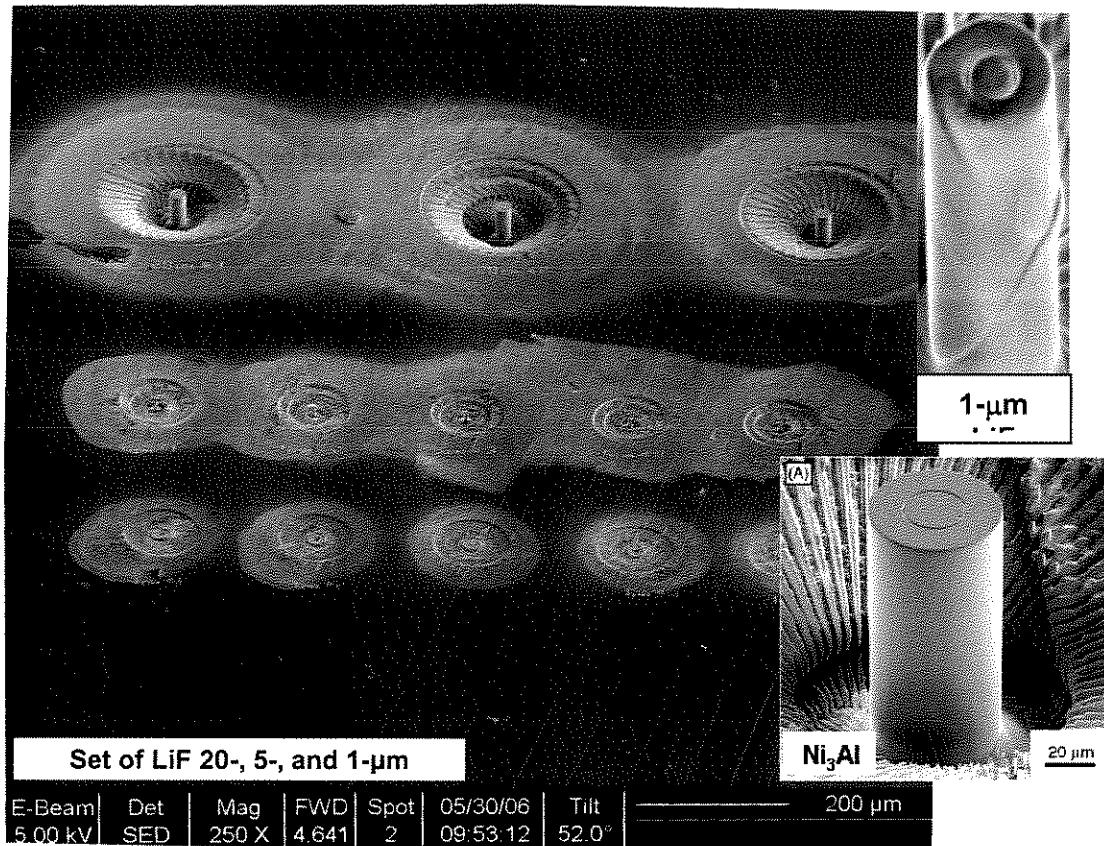


Figure 3. Set of LiF microspheres prepared by FIB technology inside an electron microscope. Also shown are 20-μm Ni<sub>3</sub>Al single crystal sample before deformation and 1- μm LiF sample after deformation. Note that only several localized slip bands can be seen on the sample surface even after as large strain as ~ 15%.

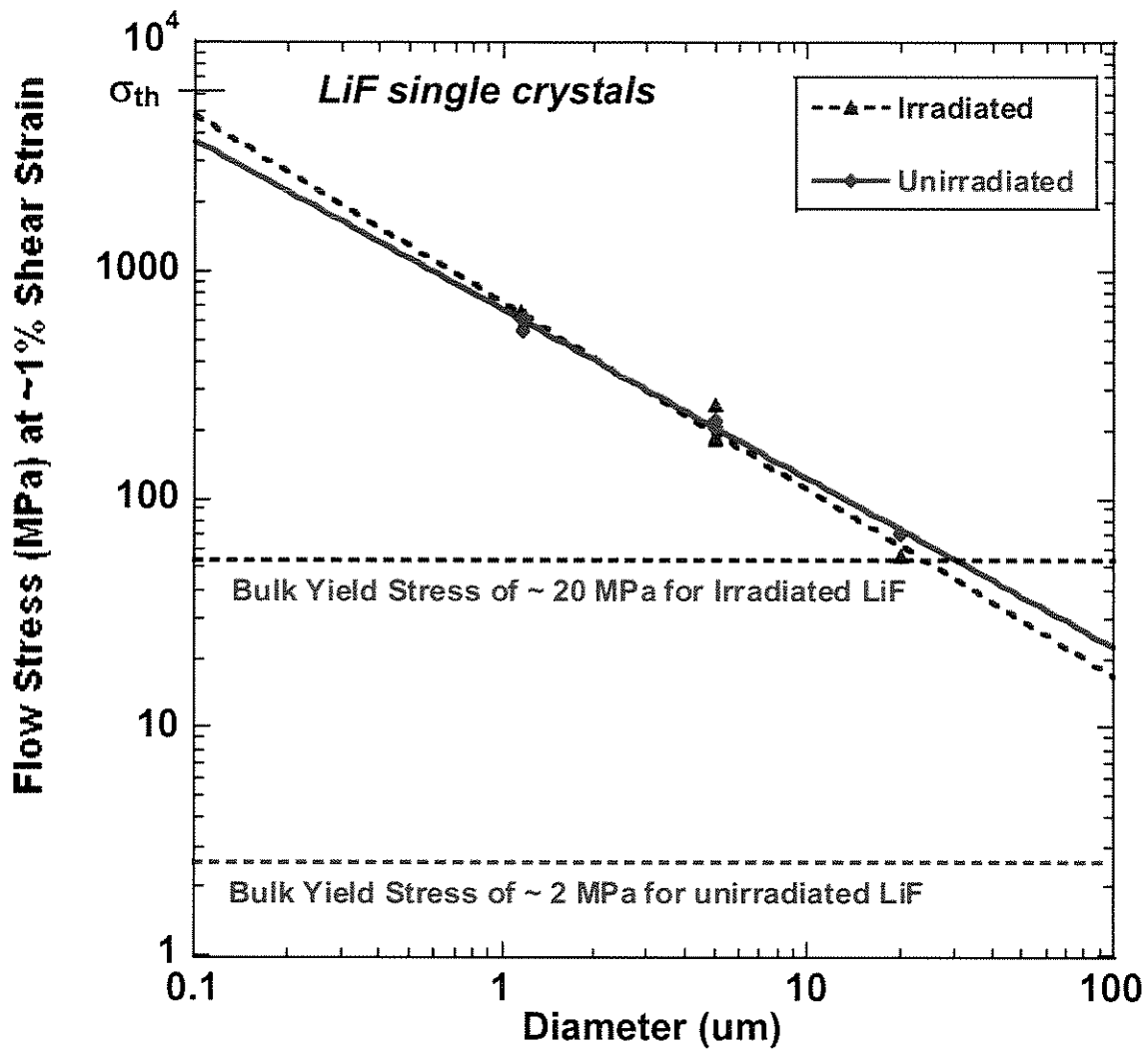


Figure 4. Size dependence in LiF single crystals: engineering-stress versus sample-diameter for 20-, 5- and 1- $\mu\text{m}$  samples. Note that despite their large yield stress difference in bulk samples, the dependences in microsamples are practically identical and could be extrapolated to values close to the theoretical strength of the given material, in this case, near 6000 MPa. However,