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Materials Processes Observed using Dynamical Environmental TEM at University of Illinois

Aram Yoon[1,2] , Wenpei Gao[1, 2], Jianbo Wu[1,2], Hyuk Park[1, 2], J. Mabon[2], W.L. Wilson[2], Jian-Min Zuo[1, 2*]
[1]Dept of Materials Science and Engineering, University of Illinois, Urbana-Champaign, IL 61801, USA
[2]Frederick Seitz Materials Research Laboratory, University of Illinois, Urbana-Champaign, IL 61801, USA

The progress in the development of dynamical environmental TEM (DETEM) at University of Illinois is reported here together with preliminary results that have been obtained in the study of nanostructure transformation and metal oxidation. The instrument development project was motivated by the need to improve time resolution in in-situ TEM study of various materials processes. TEM in general provides outstanding spatial resolution for atomic structure determination and small probes for electronic structure and chemical analysis. The use of imaging aberration corrector in an ETEM now enables atomic resolution visualization of structural transformations under variable temperatures and gas environments close to materials' real operational conditions (for a review, see ref [1]). However, the types of structural transformations that can be observed are limited by the sample stability and acquisition rate of electron images or diffraction patterns [2]. The development of MEMS based heating holders has improved the sample stability issue. For time resolution, the use of laser driven photocathodes provides superior time resolution by taking snap shots using ultrafast electron pulses [3]. While time resolution at 15 ns has been reported for recording a sequence of irreversible processes using pulsed electron beams [3], the time interval that can be recorded by this technique is currently very limited.

A Hitachi H9500 80-300kV TEM with a LaB_6 emitter is used as the development platform for DETEM. The major improvements already made include the design and installation of a gas handling and mixing system, a dual camera system with a Gatan Orius camera for video rate image recording and K2 IS camera for direct electron detection and fast recording at 400 fps for full frame transfer or 1600 fps at ¼ frame transfer [4], and a high temperature sample heating holder up to 1500 °C [5].

The improvement of time resolution was demonstrated in the heating experiment of Au catalyst on silicon nanowires (NW). At 2.5 ms apart when the Au catalyst was heated to 400 °C, initial movements of Au catalyst were observed (Figure 1 shows an example), followed by a significant change in the wetting angle of Au nanoparticle. Afterwards Au was seen diffuse away along the NW surface as indicated by fast image contrast change.

Further experiments on metal oxidation have been carried out thanks to the environmental capability of our TEM. Metal oxidation is one of the spontaneous chemical reactions. Because of its large impact on infrastructure and transportation, surface oxidation and its role on structure and property have attracted significant attentions, especially on the fundamental aspects of oxygen diffusion into the metal or metal ion transport. Figure 2 shows an in situ observation of oxidation of zircaloy-2, which is a nuclear cladding material. The experiment was carried using the special holder designed for the ETEM to provide gas injection and Joule heating directly on the sample [5]. We maintained the air pressure of $2x10^{-3}$ Pa, while heating the sample to 900°C during recording using the Orius SC200 camera (Gatan, Pleasanton, CA). The images capture the nucleation and decay of nanoparticles formed on the surface oxide of zirconium metal.

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[6] Corresponding author Jian-Min Zuo, jianzuo@illinois.edu. The instrument development is supported by the NSF MRI Grant NSF DMR 12-29454 and University of Illinois. Aram Yoon and Jianbo Wu was partially supported by DOE BES under contract DEFG02-01ER45923. Wenepi Gao was supported by NSF DMR 0449790. We thank Prof. Brent Heuser for supplying zircaloy sample and discussions.

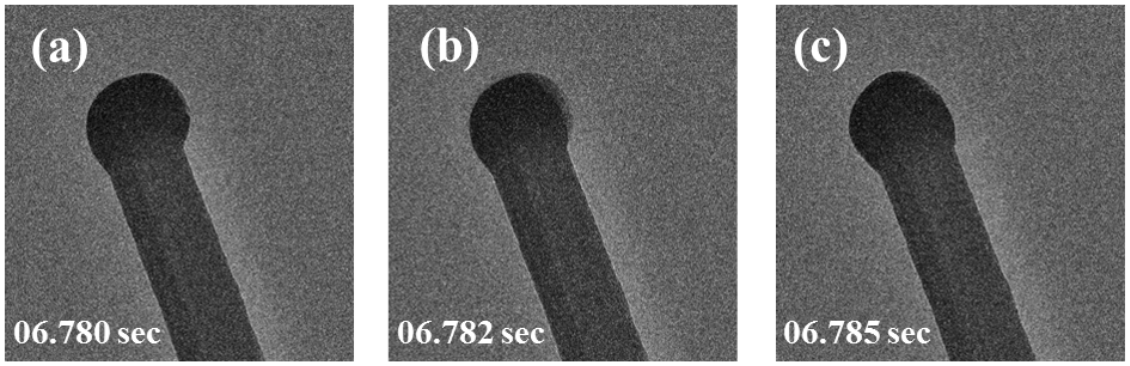

Figure 1. Au catalyst movements on a silicon nanowire captured by a fast direct detection electron camera. The time stamp below shows frames captured into 6 seconds of observation with 2.5ms time interval.

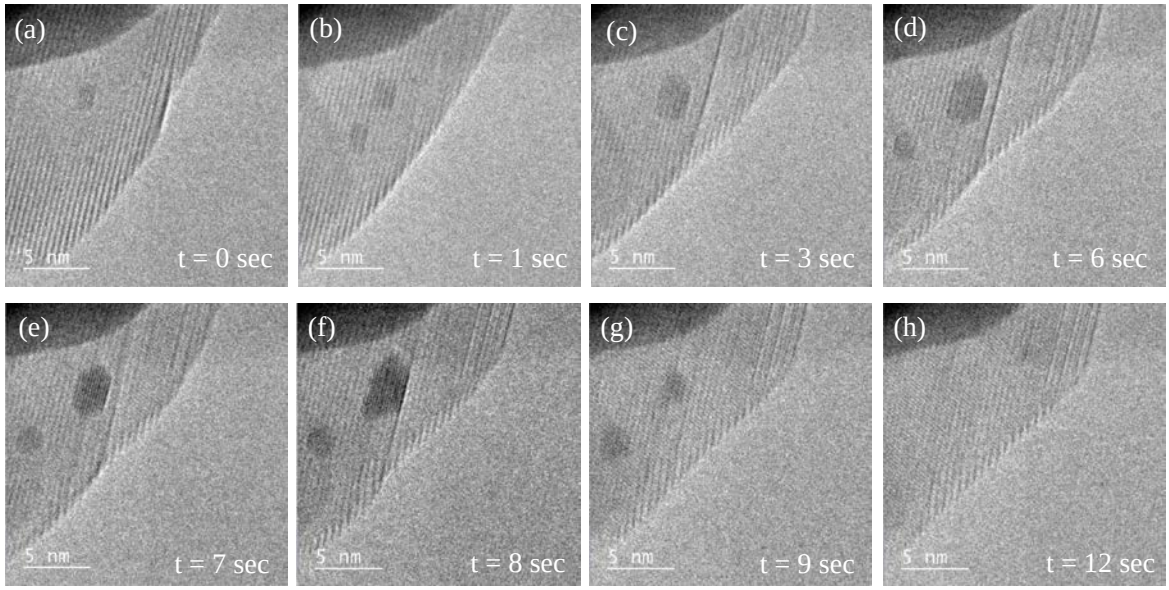

Figure 2. Oxidation of zircaloy observed at 900^0C in air with high resolution (magnification of 400K) recorded at video rate of 20 frames /second.