Thermal Expansion Coefficient Measurement from Electron Diffraction of Amorphous Films in a TEM

Hayashida, M.[1], Cui, K.[2], Malac, M.[2] and Egerton, R.[3]

[1] NRC-NANO, Canada, [2] Nanotechnology Research Center, Canada, [3] University of Alberta, Canada

Accurate knowledge of the local TEC is required by the semiconductor industry, as well as to understand the materials science of the studied materials at nanoscale. For example, TECs are used to determine if the matching between semiconductor device components and their substrates is suitable within a certain temperature range. Nanoscale measurement of a TEC is a complicated matter but can be accomplished using a transmission electron microscope (TEM). The TEM can form a small probe whose high-energy electrons interact strongly with the sample, allowing examination of a minute volume of a material. TEC measurements in amorphous materials are additionally challenging due to absence of the sharp diffraction rings or peaks that are typical in (poly) crystalline materials.

The temperature coefficient of thin films can be measured by Raman spectroscopy and electron energy-loss spectroscopy (EELS). Here we report the use of electron diffraction (ED) in a TEM to locally measure the TEC of amorphous thin films. When samples are sufficiently thin to give a TEM diffraction pattern, the TEC of polycrystalline films can be measured from the change in radius of the Bragg rings as a function of sample temperature. Many materials expand with increasing temperature, implying that the radius of diffraction rings decreases with increasing temperature. Such materials are said to have a positive TEC. Although rare, some materials have a negative TEC.

By incorporating a material with a known TEC, sample temperature can be measured locally and crystalline heavy-metal nanoparticles have been used for that purpose. The temperature was measured from the radius of diffraction rings and correlated with thermal expansion of the crystal lattice. However, the use of heavy-metal nanoparticles complicates sample preparation and can lead to contamination of the studied sample by the heavy metal.

On the contrary, the use of a standard support film, such as SiN, F/C and *a*C eliminates any need for the addition of nanoparticles during sample preparation, prevents sample contamination by the said nanoparticles and can be used for any sample compatible with the amorphous support film. We measured the linear thermal expansion coefficients of amorphous SiN and a F/C films using electron diffraction in a TEM. The broad diffraction rings of a SiN and F/C could be fitted with sufficient precision (less than 35°C RMSE) to enable the temperature coefficient of expansion to be measured, even though the ring radius changed by only a few pixels between lowest and highest temperatures. Positive thermal expansion coefficients were observed for SiN and a negative coefficient for F/C films. The low RMSE of the TEC measurement indicates the possibility of using standard SiN and F/C films for in-situ local temperature measurements in a TEM. It was also observed that TEC of SiN changes depending on window size and thickness. On the other hand, aC films cannot be used for in-situ sample temperature measurement because their ring radius continue to decrease over a few hours at a fixed temperature.