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LIMITS OF DETERMINING STRESS STATES BY FIB METHOD DUE TO GA IMPLANTATION

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In order to assess the stress state in different kinds of materials in a reliable and flexible way, a Focused Ion Beam Microscope (FIB) routine has been developed to obtain information on the stress state in a material, in particular in thin films [A-C]. This method, named FIB-DIC micron-scale ring-core method, is based on monitoring relaxation of the material during a progressively deeper milling with a FIB beam. With digital image correlation (DIC), the change in the feature position can be analyzed and converted in residual strain and then with some modelling to residual stress. The method has already been applied to a multi-phase material to map its residual stress [A]. In order to standardize this incremental FIB-DIC micro-milling routine for the evaluation of intrinsic stresses at the sub-micron scale, the limits of the method need to be studied in detail.
One of the important questions is the limitation of the technique due to Ga implantation. In the present wok we attempt to identify the zones impacted by FIB milling and the extent of Ga penetration and to determine the resolution limit of the method due to ion affected zones. Transmission Electron Microscopy (TEM) and Atom Probe Tomography (APT) are employed on a variety of materials to visualize and evaluate the Ga-induced zones. TEM is used to assess the nature and spatial distribution of the defects and to quantify the distribution of elements using Energy Dispersive Spectroscopy (EDS). APT is used to assess the spatial distribution of Ga, after its implantation into the sample via FIB and to visualize and quantify local concentration gradients and segregations that might be of interest. This approach is used on a number of materials, such as TiN, DLC, Si and a BMG. The local Ga concentration variations are analyzed as a function of ion implantation parameters, under the “normal” FIB-milling conditions (used for conventional TEM and APT specimen preparation) and also at increased Ga rates (using different Ga-source settings). As a first step, we show that the Ga implantation can be kept relatively small, when using the procedures reported in literature to reduce Ga implantation (smaller voltages, capping layers) [D, E]. Nevertheless, a change of the structure in a Si-specimen, where the slightly thicker part of a TEM lamella was still crystalline, while the thinner part transformed to a fully amorphous state, could be observed. The second step consists of preparing samples that have undergone specifically set higher Ga doses initially, but then were Ga-treated in the same gentle way, to avoid additional Ga implantation. The ultimate goal is to apply the incremental FIB method to determine stresses in the material and finally prepare site specific samples to analyze by, and correlate between TEM and APT.

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