Measurements on the nanoscale: a study of defects in thin-film FePd

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Humans have been measuring new things for thousands of year. As technology has progressed, so have the requirements on precision and being able to measure ever smaller things. For thin-film samples this can be thicknesses of a few nanometres. But how can you measure something that is ten thousand times smaller than the width of a hair? In this thesis I have used a variety of methods to measure defects this small in thin-film iron-palladium (FePd).

Defects are an (almost) unavoidable reality of crystals. They can for example affect how a material conducts electricity, its strength or its magnetic properties. Sometimes this might be positive, sometimes it might be negative, but an understanding of the effect it has is vital for an understanding of the material's behaviour.

Atomic force microscopy (AFM) is a technique for measuring the surface of a sample. In it, a cantilever with a tip is scanned over the surface, and is deflected based on the height of the surface. This can then be measured to make a map of the heights for different areas with high precision. In this thesis, AFM showed that the sample had steps on the surface, bounding large flat areas called terraces. These steps were always at a 90 ° angle from each other and 45° angle from the sample edge.

Transmission electron microscopy (TEM) is a method for measuring inside a sample with atomic-level resolution. The principle is that you have a thin slice of the sample and fire electrons through it. These electrons interact with the sample, which can be measured. Using TEM I showed that FePd had defects going through the sample all the way to the surface, where they create the surface steps that were seen in AFM.

To measure the thicknesses and roughnesses of layers in the sample, X-ray reflectometry (XRR) was employed. X-ray reflectometry exploits that when X-rays hit the surface or a layer boundary, a part will get reflected and a part will get transmitted further into the sample. Waves that are reflected at different places, such as at the surface or at a deeper layer boundary, will have travelled different distances based on the layer thickness. This difference will cause them to interfere with each other, either constructively or destructively, giving oscillations in the measured intensity. This can then be fitted to get the layer thicknesses and their roughnesses. In this thesis it was found that a very good fit could be achieved without taking the observed defects into account.

While XRR gives information about the layer structure of the sample, grazing-incidence small-angle X-ray scattering (GISAXS) can be used to get information about significant features of those layers. In XRR you only look at the reflection, that is when the incident angle is the same as the outgoing angle. In reality, the X-ray can also get scattered, both to the side as well as upwards and downwards. This can be seen in GISAXS, which allows for modelling of what might cause the scattering. I found that the sample produces two "arms" that are sensitive to the rotation of the sample. These arms could be reproduced in modelling by using pyramids to represent the surface steps.

To summarise, the measurements of the FePd sample showed that there were defects in the sample, leading to steps on the surface. These steps did not affect the XRR measurements, but when modelling the GISAXS, they have to be taken into account.