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SEMINAR

Monolayer quantification using wavelength dispersive X-Ray spectroscopy

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Abstract

Electron probe microanalysis (EPMA) using wavelength dispersive X-ray spectroscopy (EPMA-WDS) is applied to the quantification of surface and grain boundary monolayer segregation. The case of sulphur segregation in nickel and nickel alloys is considered. It is evidenced that EPMA-WDS is able to detect submonolayer surface segregation. The sulphur segregation can be accurately quantified from the sulphur Ka line relative intensity (ratio of the intensity measured on the sample and the intensity measured on a standard material) using the StratagemÔ software (analytical modelling of the X-ray emission in a stratified specimen based on the PhiRoZ model). The statistical accuracy of the technique and its detection limit are estimated to be as low as a few percents of a monolayer for reasonable counting times (~ a few minutes). The advantages and drawbacks of EPMA-WDS with respect to Auger electron spectroscopy (AES) are discussed. The main advantage of EMPA-WDS is that it is almost insensitive to surface contamination and oxidation, which makes it possible to measure surface segregation on samples that have been in contact with atmosphere. The influence of specimen tilt is also discussed.

The technique is also applied to the measurement of sulphur segregation on the fracture surface of an iron-nickel alloy sample broken at high temperature. It is thus demonstrated that EPMA-WDS could be a very useful tool for failure analysis in the case of grain boundary fractures.

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