Acta Cryst. (2002). A**58** (Supplement), C26

SMALL-ANGLE SCATTERING, A TOOL TO CHARACTERIZE Ni-BASE SUPERALLOYS AT HIGH TEMPERATURE

R. Gilles[1] P. Strunz[2] D. Mukherji[3] S. Roth[4] A. Wiedenmann[5] J. Roesler[3] H. Fuess[1]

[1]TU Darmstadt, Petersenstr. 23, 64287 Darmstadt, Germany [2]Nuclear physics Instiute, 25068 Rez near Prague, Czech Republic [3]TU Braunschweig, Langer Kamp 8, 38106 Braunschweig, Germany [4]ESRF, 38043 Grenoble, France [5]Hahn-Meitner-Institut Berlin, Glienicker Str. 100, 14109 Berlin, Germany

Small-angle scattering (SAS) technique with neutrons and X-ray facilitates detailed characterization of precipitates and other inhomogeneities in Ni-base superalloys. The control of the morphology and distribution of γ'-precipitates is essential for high-temperature strength of components (e.g. turbine blades) made of these materials. A traditional way to investigate the kinetics of precipitate dissolution and growth is through microstrutural observations on samples quenched from high temperatures. The advantage of SAS method is to watch in situ the changes in morphology at the temperature. Small-angle neutron scattering (SANS) experiments with easy handling of relative large sample volumes and flexible sample environment allow the bulk investigation, whereas the synchrotron microbeam (M-SAXS) measurements provide very local information. Several SANS experiments (V4) at HMI Berlin, using Re-rich superalloys (both polycrystalline and single-crystal) to investigate γ'-solutionizing and coarsening are presented in this contribution. Such experiments are intended to understand the evolution of microstructure at elevated temperatures as well as to help to optimize homogenization and final microstructure of the superalloy. Another important factor influencing the mechanical properties is the presence of topologically close packed (TCP) phases, which can be detrimental for the mechanical properties of superalloys. They are easily detected by SANS in spite of their low volume fraction. Nevertheless, a detailed local study of such oriented TCP precipitates and their surrounding can be advantageously done with help of M-SAXS as shown by the last experiment performed at ESRF (ID13).

Keywords: SMALL-ANGLE SCATTERING, SUPERALLOYS, PRECIPITATES

Acta Cryst. (2002). A**58** (Supplement), C26

X-RAY COMPLEX ON THE BASIS OF KUMAKHOV CAPILLARY OPTICS FOR MICROELECTRONICS INVESTIGATIONS

N. Ibraimov[1] S.V. Nikitina[2] O.V. Mikhin[3] A.V. Mozhaev[4] D. Malagodi[5] S. Srinivasan[6]

Institute For Roentgen Optics 1st Volokolamsky Pr. 10, PMZR Moscow 123060 RUSSIA

An X-ray complex has been developed consisting of three functional assembly units. On the basis of Kumakhov's optics, a block diagram of X-ray reflectometer has been developed for roughness control of polished surfaces and analysis of thin films used in microelectronics. The device includes a point-focus tube of 2 W power, X-ray half-lens, flat monochromator SiO_2, scintillation detector. Capillary half-lens produces spatially collimated X-ray, permitting a considerable reduction in instrument dimensions and X-ray tube power compared to the existing models. The instrument's optical efficiency is 5-6 times higher than when Soller slits are used. Capability of Kumakhov's half-lenses to raise the density of collimated X-ray quanta enables a significant improvement with regard to optical efficiency and resolution of the instrument, as well as background noise reduction.

Spectral assembly permitting simultaneous analysis of the elemental composition of the samples under analysis using incident K α monochromatic beam at a close-to-critical angle. Low-angle incident X-ray beam, ~25 micron wide, allows to increase the effective area of excited fluorescence that is registered by the energy dispersive detector. Double crystal spectrometry assembly is completed with a set of Si and Ge monochromators permitting dispersion-free rocking curves for any Bragg's angles without additional alignment of the instrument and providing for the following measurements: 1. Assess crystal structure perfection of wafer surfaces after various technological processes. 2. Measure film-wafer Δd/d during epitaxy. 3. Measure parameters of multi-layer coating super-structure.

Keywords: REFLECTOMETRY, THIN FILM, SURFACE ANALYSIS

Acta Cryst. (2002). A**58** (Supplement), C26

ENHANCEMENT EFFECTS OBSERVED IN GRAZING-INCIDENCE NON-SPECULAR X-RAY REFLECTION FROM MULTILAYERS

K. Sakurai S Kuwajima

National Institute For Materials Science 1-2-1, Sengen TSUKUBA IBARAKI 305-0047

Specular and non-specular x-ray scattering observed at grazing incidence is a powerful tool for investigating the morphology of interfaces of thin films as well as the surface. It is possible to determine the roughness, compositional/density gradients, horizontal and vertical correlation length, and the hurst parameter. However, in practice, it is not quite so easy to obtain a good reasonable fit when the number of parameters increases as in the case of multilayers. Although uniform correlation along the depth is not realistic, it is frequently assumed as constant to simplify the model. On the other hand, interference effects generally observed for thin films is feasible for exploring specific portion of the layered structure. In the case of specular reflection, appearance of the fringes and/or bragg peaks for periodic multilayers is widely used for determining the distances between layers. Similarly, fringes and/or "bragg" peaks observed in non-specular scans provide information on the correlation of interfaces. Experiments have been performed with 16 kev x-rays at bl-14a, photon factory. The samples measured are w/b4c (2d = 3.9 nm) and some other multilayers. Strong enhancement effects have been observed when qz is close to the bragg condition. They have been often qualitatively interpreted as conformal roughness, but are also affected by the enhancement of the electric field intensity. In the present paper, the effects have been investigated in detail experimentally, and are quantitatively discussed in terms of the interfaces with variable vertical correlation.

Keywords: INTERFACE ROUGHNESS THIN FILMS

Acta Cryst. (2002). A**58** (Supplement), C26

CAUGHT IN THE ACT OF CATALYSIS

I. Schlichting

Max Planck Institute for medical Research, Dept. of Biomolecular Mechanisms, Jahnstr. 29, 69120 Heidelberg, Germany

In the general excitement of a time when new protein structures are published every week, it is often forgotten that a structure in itself does not tell one how the molecule works. For that, one needs to know a great deal about mechanism, intermediates, and dynamics. Owing to the large gap between the time scales of protein motions or enzymatic reactions on the one hand, and crystallization and data acquisition on the other hand, crystallographic determination of structures of reaction intermediates is usually not straightforward.

In the underground of macromolecular crystallography is a cohort of investigators working to understand the time course of reactions mediated by these large molecules. Over the years methods to initiate reactions in crystals have been developed and there has been success from a variety of approaches including Laue crystallography and freeze trapping of intermediates. We'll review these strategies, describe some recent results, and provide an outlook to future developments.

Keywords: REACTION INTERMEDIATES TRAPPING LAUE