C03A16XU-3018-N BL16XU

High Energy X-ray Diffraction Analysis of Protective Coating

Hiroshi Deguchi (4133)[1,*], Masaki Horiuchi (4926)[2], Masato Oshitani (6993)[2], Tohru Yamamoto (3959)[3], Shin-ichi Noguchi (4618)[4]

[1] The Kansai Electric Power Co. INC., [2] Kanden Kakou Co., Ltd., [3] Central Research Institute of Electric Power Industry, [4] Techno Service Co., LTD.

MCrAlY-type alloys (M=Co and/or Ni) have been successfully used as overlay coatings on gas turbine blades to protect against oxidation and hot corrosion. However, unfortunately, cracking of the coating itself often occurs during operation by stress due to a steep temperature gradient and mismatch in thermal expansion coefficients between the coating and substrate. In this study, we tried to evaluate the residual stress in the coating.

Two specimens were prepared by CoCrAlY plasma spraying on Ni base super alloy substrates. One specimen was exposed to high temperature (about 950℃), and the other specimen to low temperature (about 800℃), for 20,000 hours. The thickness of the coating was 250μm. As many long parallel cracks were observed at the high specimen surface, the stress measurements were performed in both parallel and perpendicular direction to the cracks. Though no crack was observed in the low specimen, similar bidirectional measurement was applied.

Incident X-ray of 30 keV was collimated down to 1 mm×1 mm by an incident slit. A solar slit was used as a receiving slit. 440 peak of CoCrAlY fcc phase was used for stress measurements.

Figures 1 and 2 show 2θ-$\sin^2\Psi$ plots of low and high specimen, respectively. 2θ value is different between low and high specimen because of variation in element composition caused by atomic diffusion during high temperature exposure. As for the low specimen, 2θ -$\sin^2\Psi$ plots in parallel and perpendicular direction agree well. So the stress state can be regarded as equiaxial. However, as for the high specimen, 2θ in perpendicular direction is higher than in parallel direction when $\sin^2\Psi > 0.7$. It can be suggested that the crack occurrence made the lattice spacing in perpendicular direction narrower than in parallel direction.

Further analysis is now under progress.

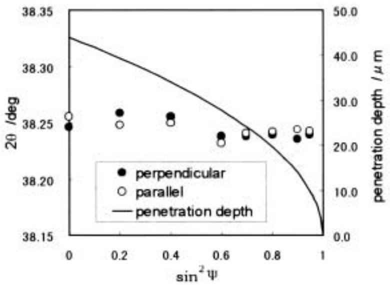

Figure 1 2θ-$\sin^2\Psi$ plot of low temperature specimen.

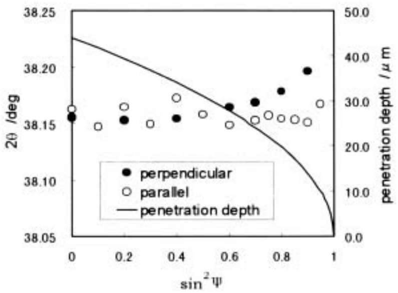

Figure 2 2θ-$\sin^2\Psi$ plot of high temperature specimen.

C03A16XU-3019-N BL16XU

Crystallite Size and Strain Analysis of Multilayer Thin Films

K. Ueda(5099)*, T. Hirano(5097) and Y. Hirai(4083) [1)]

Hitachi Research Laboratory, Hitachi, Ltd.

[1)] Advanced Research Laboratory, Hitachi, Ltd.

Giant magnetoresistive (GMR) heads have attracted several theoretical and experimental works to obtain higher areal density for hard drive. The structure of the GMR sensors consists of two ferromagnetic layers and one noble metal separator with a thickness of a few nm. Their magnetic properties depend on the grain size and the strain of the magnetic layers. Therefore, analysis of the size and strain is important for producing good sensors.

The layers of the sample, (anti-ferro, pinned-ferro, noble metal, free-ferro magnetic and cap layers) were deposited on the underlayers in turn. The anti- ferromagnetic layer is made of MnPt and the ferromagnetic ones are fcc CoFe and NiFe. An oxide layer is inserted into the pinned-ferro magnetic layers. The oxide layer controls the grain size of the magnetic layers[1)].

The in-plane diffraction patterns of the sample were measured by a diffractometer at BL16XU in SPring-8[2, 3)] with an incident x-ray energy of 12.4 keV.

The fcc(220) and (440) diffraction peaks were fitted by a Voigt function to obtain the peak width. The average crystallite size and the strain were analyzed from scattering vector dependence of the peak width (Hall's method).

Fig.1 shows the diffraction intensity (left axis) and penetration depth of x-rays into the sample (right axis) as a function of incident angle. Fig.2 shows crystallite size and strain versus penetration depth. This result indicates that both crystallite size and the strain of the free-ferro magnetic layers can be analyzed by small incidence angle of the in-plane XRD.

1) K. Ueda *et al.*; "Spring-8 User Experimental Report No.9", Jpn. Syn. Rad. Res. Ins., 285 (2002)

2) Y. Hirai *et al.*; Proc. Ann. Meeting Jpn. Soc. Syn. Rad. Res., 8P-70 (2000)

3) M. Takahashi *et al.*; Proc. Ann. Meeting Jpn. Soc. Syn. Rad. Res., 9P-67 (2000)

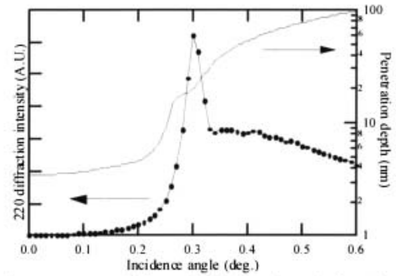

Fig.1. (220) diffraction intensity (left axis) and penetration depth of x-rays into the sample versus incident angle.

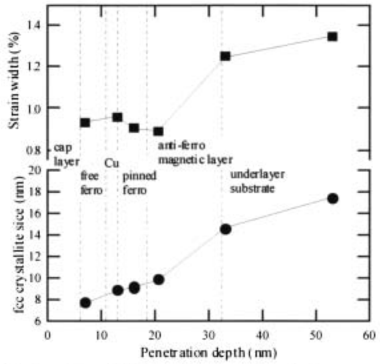

Fig.2. Crystallite size and strain versus penetration depth. Line for the eye guide.