MICROSTRUCTURE CHARACTERIZATION OF A NITRIDED FE-3WT.%CR-0.3WT.%C MODEL ALLOY BY ANOMALOUS SMALL ANGLE X-RAY SCATTERING.

Sébastien Jégou[1], Myriam Dumont[2], Laurent Barrallier[1]

[1] Arts et Métiers ParisTech, MSMP laboratory;
2 cours des Arts et Métiers; Aix-en-Provence, 13617, France
[2] Aix-Marseille Université, CNRS, IM2NP UMR 7334; Campus de Saint-Jérôme, Avenue Escadrille Normandie Niemen, Case 142 ; Marseille, 13397, France

Keywords: Steel, Nitriding, Anomalous small angle X-ray scattering.

Abstract

Nitriding is a thermo-chemical surface treatment of steels providing an improved fatigue and wear resistance. This treatment is based on nitrogen diffusion involving the precipitation of nano-scaled nitrides from the solid solution at the near surface of the nitrided piece. Nitriding involves a complex microstructural evolution both in time and depth including diffusion of nitrogen and precipitation of nitrides but also coarsening and dissolution of carbides resulting in diffusion of carbon. However the chemical composition of nano-scaled precipitates remains controversial, in particular regarding the iron content in nano-nitrides that may substitute alloying elements. In this framework, anomalous small-angle X-ray scattering was used to bring quantitative data on the distribution and composition of the nano-scaled phases in a Fe-3Cr-0.3C steel as a function of depth.

Introduction

Nitriding of steels involves a complex microstructural gradient both in time and depth including diffusion of nitrogen and precipitation of nano-nitrides but also coarsening, dissolution and re-precipitation of carbides accompanied by transverse diffusion of carbon [1,2].

Despite of the large characterization of nitrided surface, the chemical composition of nano-scale precipitates remains controversial. The purpose of this study is to bring some new insight on the composition of the nano-scale phases in different nitriding conditions, as a function of depth and therefore to provide quantitative data on nitrides in the diffusion layer essential for further understanding and modelling of the nitriding process.

Experimental method

Small-angle X-ray scattering is a powerful technique for investigation of nanometric particles and provide statistical data. Moreover SAXS carried out in the anomalous mode at the Cr-edge can provide information on the Cr-composition of the nano-nitrides [3]. Experiments were carried out on the BM02-D2AM, a French CRG beamline at the European Synchrotron Radiation Facilities (ESRF) in Grenoble. Two samples were studied with the same composition (0.35 wt.% C, 1.0wt% Cr, Fe balance) and two different nitriding conditions (10 or 100 h at 550 °C).

Main results

The evolution of the integrated intensity Q_0 as a function of depth was evaluated and is displayed in figure 1(a) for the 10h@550°C nitrided sample. The figure clearly renders the three expected zones, i.e. the compound layer close to the surface, the diffusion layer characterized by the precipitation of nitrides and the bulk material away from the surface. For each point, the Cr-content can then be determined by the analysis of the anomalous effect under the hypothesis that precipitates are MN nitrides (M=Cr or Fe). It can be shown [3] that the square root of the integrated intensity is expected to vary linearly with the atomic scattering factor of Cr, f_{Cr}, as $\sqrt{Q_0} = p\, f_{Cr} + \theta$, where p and θ can be related to the Cr-content in the precipitates :

$$\frac{\theta}{p} = \frac{\alpha\left(0.5 f_N + f_{Fe}\left(0.5 - X_{Cr,ppt}\right)\right) - f_{Fe}}{\alpha X_{Cr,ppt}}$$

where α is a constant, f_N and f_{Fe} are the atomic scattering factors of N and Fe respectively and $X_{Cr,ppt}$ is the atomic fraction of Cr in MN nitrides.
Such mathematical treatment was carried out and the resulting Cr-content in the nitrides was determined and is displayed in Figure 1(b).

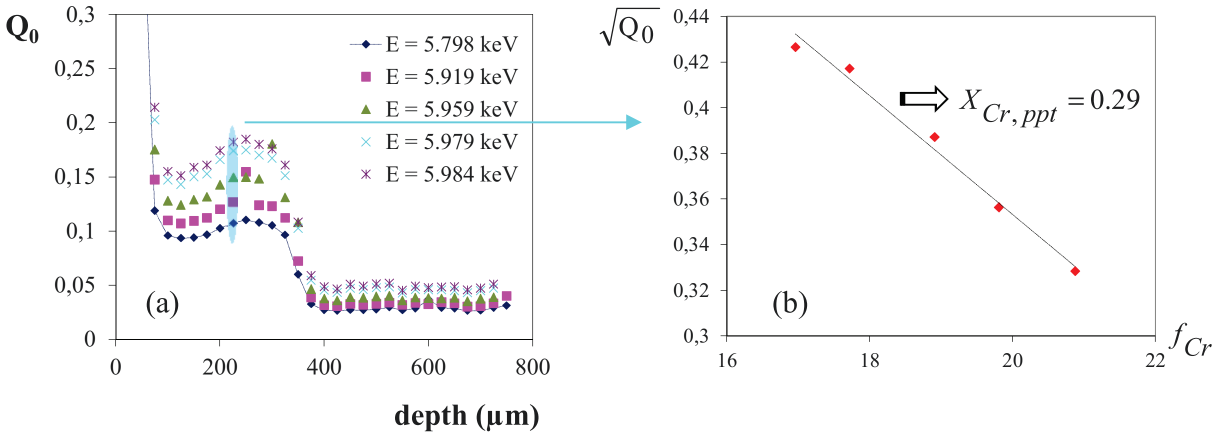

Figure 1: Evolution of the integrated intensity as a function of depth in the 10h@550°C sample (a) and linear treatment of the anomalous effect in order to extract the Cr composition (b).

Once the composition of the nitrides determined, the measured Q_0 values can be used to calculate the volume fraction of nitrides formed in the diffusion layer. It is then possible to determine in a quantitative manner the evolution of the volume fraction of nitrides as a function of depth.
Finally by using mass balance considerations, the amount of N and Cr incorporated in the nitrides can be calculated and compared to the total amount of N and to the nominal Cr composition of the steel respectively. This has been done and results give composition very close to the overall content of these elements.
The ASAXS results on two nitrided conditions of a Fe-3wt.%Cr-0.3wt.%C model alloy gives an estimation of the composition of MN nitrides formed in the diffusion layer: $Cr_{0,6\pm10\%}Fe_{0,4\pm10\%}N$. A further treatment enables to draw the quantitative evolution of the volume fraction of nitrides along the depth of the diffusion layer. This provides essential data for modelling of the microstructural evolution during nitriding and thus resulting properties.

References

1. P.C. Van Wiggen, H.C.F. Rozendaal, and E.J. Mittemeijer, "The nitriding behavior of iron-chromium-carbon alloys", *Journal of Materials Science*, 20 (1985), 4561-4582.
2. S. Jegou, L. Barrallier, and R. Kubler, "Phase transformations and induced volume changes in a nitride ternary Fe-3%Cr-0.345%C alloy", *Acta Materialia*, 58 (2010), 2666-2676.
3. M. Dumont et al., "Chemical composition of nano-phases studied by anomalous small-angle X-ray scattering: Application to oxide nano-particles in ODS steels", *Materials Characterization*, 87 (2014), 138-142.

Microstructure Characterization of a Nitrided Fe-3wt.%Cr-0.3wt.%C Model Alloy by Anomalous Small Angle X-ray Scattering: *Myriam Dumont*[1]; Sébastien Jegou[2]; Laurent Barrallier[2]; [1]IM2NP-UMR 7334; [2]MSMP

Nitriding is a thermo-chemical surface treatment of steels providing an improved fatigue and wear resistance. This treatment is based on nitrogen diffusion involving the precipitation of nano-scaled nitrides from the solid solution at the near surface of the nitrided piece. Nitriding involves a complex microstructural evolution both in time and depth including diffusion of nitrogen and precipitation of nitrides but also coarsening and dissolution of carbides resulting in diffusion of carbon. However the chemical composition of nano-scaled precipitates remains controversial, in particular regarding the iron content in nano-nitrides that may substitute alloying elements. In this framework, anomalous small-angle X-ray scattering was used to bring quantitative data on the distribution and composition of the nano-scaled phases in a Fe-3Cr-0.3C steel as a function of depth.