About some properties of amorphous gadolinium-iron thin films

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Résumé. — Des films minces amorphes d'alliages de gadolinium et de fer de compositions très variables sont préparés par évaporation sous vide. Sur des échantillons bien caractérisés (structure, composition, épaisseur, homogénéité), des mesures de spectres d'énergie ont permis de déterminer le spectre des excitations électroniques entre 5 et 150 eV et des observations en microscopie de Lorentz d'étudier la distribution locale de l'aimantation.

Abstract. — Amorphous thin foils of Fe-Gd alloys with very different compositions are prepared by vacuum evaporation. On well characterized specimens (structure, concentration, thickness, homogeneity), energy loss measurements have led to the determination of the electron excitation spectrum between 5 and 150 eV and Lorentz microscopy observations have been used to visualize the local magnetization distribution.

The purpose of this paper is to describe some electron microscopy studies concerning a family of Gd-Fe thin foils (i.e. with thickness below 100 nm) of quite varying compositions. Important physical features such as the crystallographic structure (as seen by electron diffraction), the chemical composition (checked either by X-ray microprobe or electron energy loss spectroscopy), the magnetization (revealed by Lorentz microscopy), the homogeneity... can thus be observed on a spatial extension of the order of one micron or less. This approach provides a local characterization of the specimen properties, rather different from other generally used techniques (either spectroscopic or magnetic) which average the results over specimen volumes several orders of magnitude larger.

For this work, specimens have been prepared over wide composition ranges by vacuum evaporation on substrates maintained at room temperature and two slightly distinct methods have been used :

a) Joule preparation from a conglomerate made of scraps of rare-earth metal and powder of transition metal. A preliminary heating between 1 150 and 1 500 K forms an homogeneous compound which is then evaporated at higher temperature (about 1 800 K). Foils with thickness ranging from 30 to 300 nm are deposited with rates between 2 and 100 nm/s.

b) Scanning electron-beam evaporation from two crucibles containing the pure elements. In a high vacuum chamber (10^{-10} torr) an electron beam can be focused either on one or the other of two crucibles, the scanning of the beam being governed by a signal whose frequency and dwelling time can be adjusted continuously. Thin films of about 50 to 100 nm are deposited with rates between 0.1 and 1.5 nm/s.

Both types of specimens generally exhibit an electron diffraction pattern with diffused rings which suggest an amorphous state. It will be mainly dealt in this paper with foils prepared following mode *b*) because X-ray microprobe measurements show that the at. % of Gd is roughly proportional to the percentage of focusing time of the electron beam on the Gd crucible during the evaporation.

1. **Energy loss functions.** — It is well known that the energy loss function (and the dielectric or optical constants) calculated from the energy loss spectrum of high energy primary electrons transmitted through thin specimen foils can reveal the important contributions in the excitation spectrum. Such an analysis, which had been achieved on pure rare-earth metals [1], is now extended to the case of alloys. Results are shown in figure 1 for three specimens of different chemical compositions. The main characteristics are the following :

— a shift and a change of the plasmon loss profile from 15 eV for the Gd rich foil to 23 eV in the Fe rich one. The effective number of *valence electrons* involved in excitation processes over the energy range 0-50 eV therefore includes the 3d transition electrons but very little the 4f rare-earth electrons ;

— the occurrence of well defined core-loss signals due respectively to the O_{2-3} (at 35 eV) and N_{4-5} (at 155 eV) edges in Gd and M_{2-3} (at 55 eV) edges in Fe. These lines constitute a useful way for the

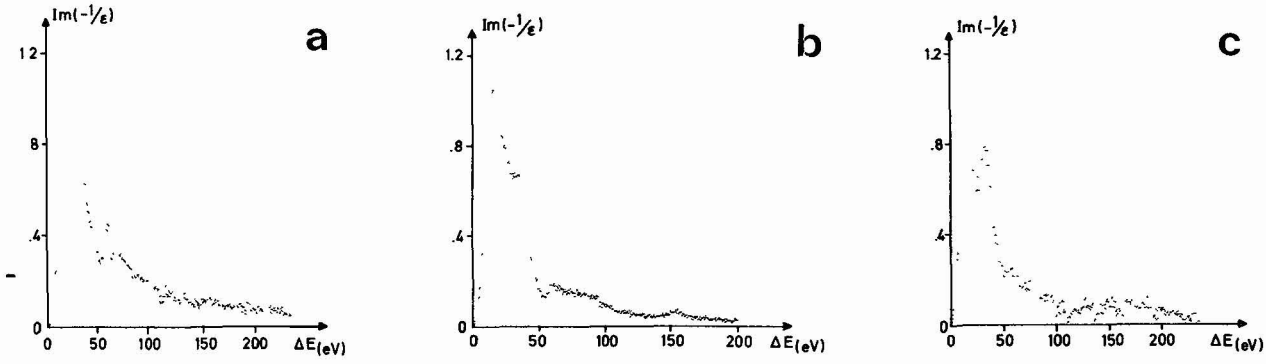

Fig. 1. — Energy loss function from Gd_xFe_{1-x} alloys of different composition. *a*) $x = 0.05$, *b*) $x = 0.25$, *c*) $x = 0.65$.

estimation of the elemental composition. Moreover energy loss spectra are actually recorded on areas of 0.05 μ^2 instead of 25 μ^2 for a conventional microprobe analysis. Composition inhomogeneities have been detected at this lower scale (below 1 μ in extension) which cannot be revealed by X-ray measurements over typical 5 μ distances.

It is possible to deduce from the measurement of the energy loss function between 2 and 100 eV an evaluation of the local thickness of the foil [2] with an accuracy of $\pm$ 10 % on the range 30 to 100 nm. This method has been tested on various standards ((Au and C)) and has been used in the present work for an *a posteriori* determination of the local thickness.

2. **Lorentz microscopy.** — The magnetic domain configuration can easily be visualized in an defocused electron microscope image (Lorentz microscopy). This technique has been extensively used for the observation of magnetic patterns in all the specimens before and after the application of an external perpendicular magnetic field greater than the field necessary to reach saturation.

a) For the thin specimens prepared by electron-beam scanning evaporation, the magnetization remains generally in the plane of the foil for all the studied compositions (between 5 and 65 % of Gd). This is revealed by a characteristic magnetic structure with clear domain walls as shown in figure 2*a*. The $M - H$ loops confirm this behaviour. Only in some

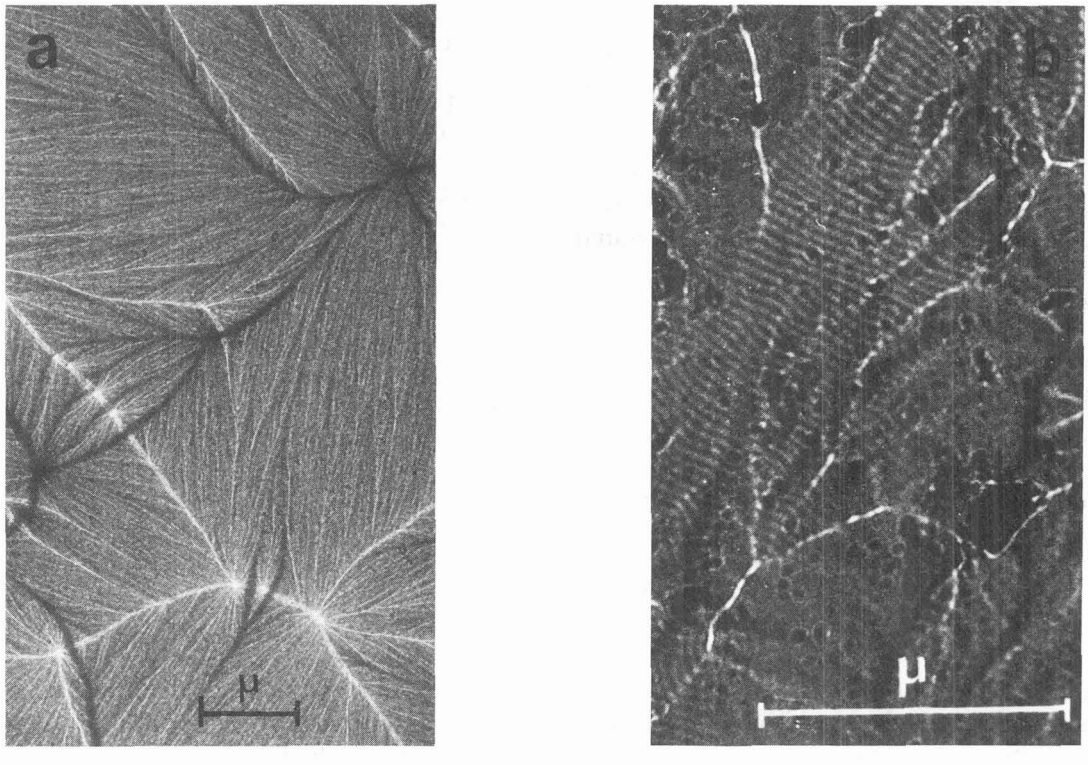

Fig. 2. — *a*) Domain wall structure generally observed in freshly electron beam deposited foil, corresponding to an in-plane magnetization ; *b*) Stripe domains observed in a reduced area of one of these Gd_xFe_{1-x} foils with $x \simeq 0.25$.

restricted areas, stripe domains (such as those visible in figure 2*b*) could be detected for a composition close to 25 % of Gd.

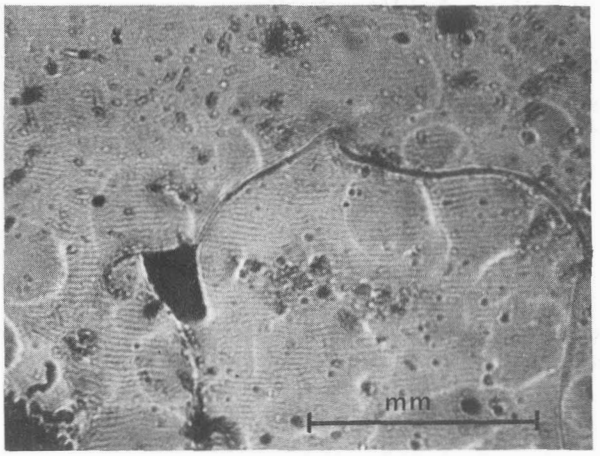

Fig. 3. — Bitter figure of a Joule deposited foil (see text).

b) For the Joule deposited specimens no systematic study of the magnetic behaviour could be developed. However, in a few cases, a magnetization perpendicular to the foil plane has been detected either through a square $M - H$ loop from Hall effect measurements, or from the observation of regular stripe domains by Bitter technique. An example is shown in figure 3 concerning a foil 260 nm thick, with a composition close to 25 % Gd and a temperature of reversal of the Hall polarity at 255 K.

In the present state of our study, the specific characters of the Joule technique involving a higher concentration of impurities and a quicker rate of growth could be responsible of this perpendicular anisotropy.

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