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# Time effects in the thermal annealing of Fe/V multilayers

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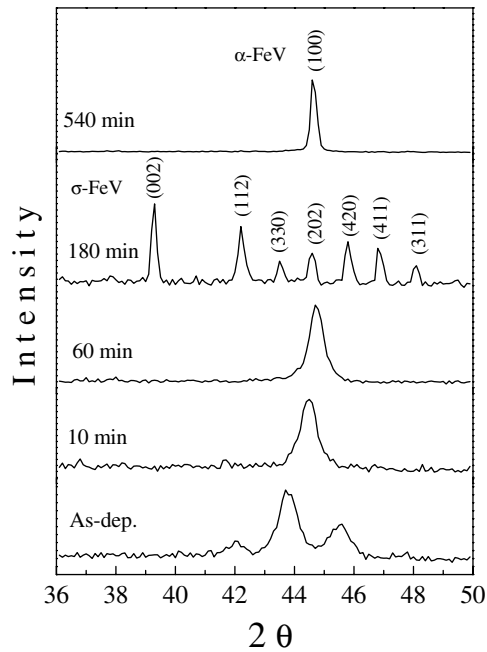
## Abstract

We report a study on the structural and magnetic properties of iron–vanadium thin films grown in multilayer form and mixed by thermal treatment. The multilayer samples were annealed at 610 °C for times ranging from 10 to 540 min. The samples were structurally characterized by means of x-ray diffraction (XRD) and by x-ray absorption spectroscopy (XAS). The magnetic characterization was carried out with a conventional alternating gradient magnetometer (AGM) and by conversion electron Mössbauer spectroscopy (CEMS). The XRD result for the as-deposited multilayer shows a high degree of crystallinity while the CEMS result suggests an abrupt interface, since no significant contribution from vanadium in iron is observed. After the thermal treatment, the results from XRD show a phase transformation of the disordered body-centred-cubic structure ( $\alpha$ -phase) into a tetragonal structure ( $\sigma$ -phase) and a subsequent return to the  $\alpha$ -phase. This  $\alpha$ – $\sigma$ – $\alpha$  oscillation is not reported in the literature available to the authors.

## 1. Introduction

Magnetic solid solutions of Fe and V have been studied for years, and electronic structure calculations indicate a reduction of the Fe atomic magnetic moment when an ordered phase is obtained [1]. Great attention has been devoted to the study of interface and surface magnetic anisotropy and to the analysis of the interplay between magnetoresistance and magnetic coupling of ferromagnetic layers separated by noble metal spacers [2]. Vanadium, which is nonmagnetic in bulk form, apparently acquires an induced magnetic moment when forming a multilayered structure of Fe/V or when it is grown as an ultrathin film on single-crystalline

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**Figure 1.** XRD of samples subjected to thermal treatment at 610 °C for several durations.

Fe substrates [3], and recently these studies have been extended to alloys such as  $\text{Fe}_{1-x}\text{V}_x$  [4–6]. Regarding the structural aspect, the Fe–V phase diagram displays a continuous high-temperature bcc solid solution intersected at temperatures below 1250 °C by a tetragonal  $\sigma$ -phase centred on equiatomic composition [7]. Due to the very sluggish character of the bcc-to- $\sigma$  transformation, it is possible to retain the bcc solid solution at low temperatures, and this metastable solid solution has a tendency to order with a CsCl-type structure [8]. Fe–V alloys in the disordered bcc  $\alpha$ -phase, in the ordered CsCl-like structure, and in the tetragonal phases have been obtained by thermal treatments of Fe/V multilayers [9]. For temperatures up to 610 °C the multilayer structures are destroyed and formation of a cubic ferromagnetic phase is observed. For temperatures above 610 °C an ordered bcc  $\alpha'$ -phase occurs and above 640 °C a tetragonal  $\sigma$ -phase is obtained, both with a decrease of the Fe magnetic moments. In this paper we report a study of the time evolution of thermal treatments at 610 °C of Fe/V multilayers. The system has been investigated using x-ray diffraction (XRD), x-ray absorption spectroscopy (XAS), magnetization, and conversion electron Mössbauer spectroscopy (CEMS).

## 2. Experimental details

Samples of  $[\text{Fe}(20 \text{ \AA})/\text{V}(20 \text{ \AA})]_{20}$  multilayers were prepared by alternating deposition in ultrahigh vacuum on commercial oxidized Si(111) substrate at room temperature. We used the electron beam technique in a BALZERS UMS-500P system at  $1 \text{ \AA s}^{-1}$  controlled by a quartz microbalance. The total nominal thickness of the films was 800 Å and the thickness of the layers was chosen to yield a 50–50 concentration. The samples were thermally treated at 610 °C for 10, 60, 180, and 540 min at a pressure of  $10^{-6}$  mbar. After the thermal treatments, the films were cooled down to room temperature in vacuum. The structure of the samples was characterized by XRD in the  $\theta$ – $2\theta$  Bragg–Brentano geometry using Cu  $K\alpha$  radiation

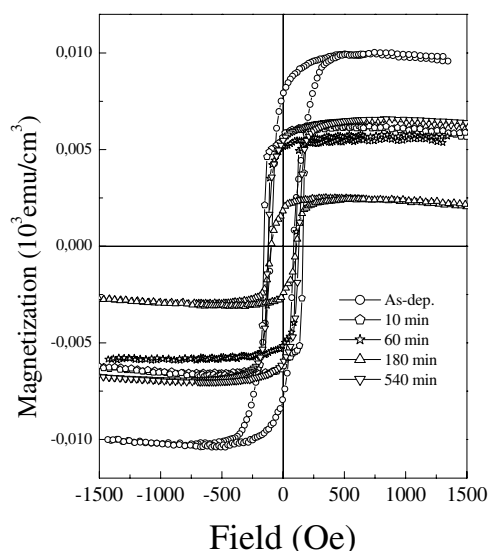
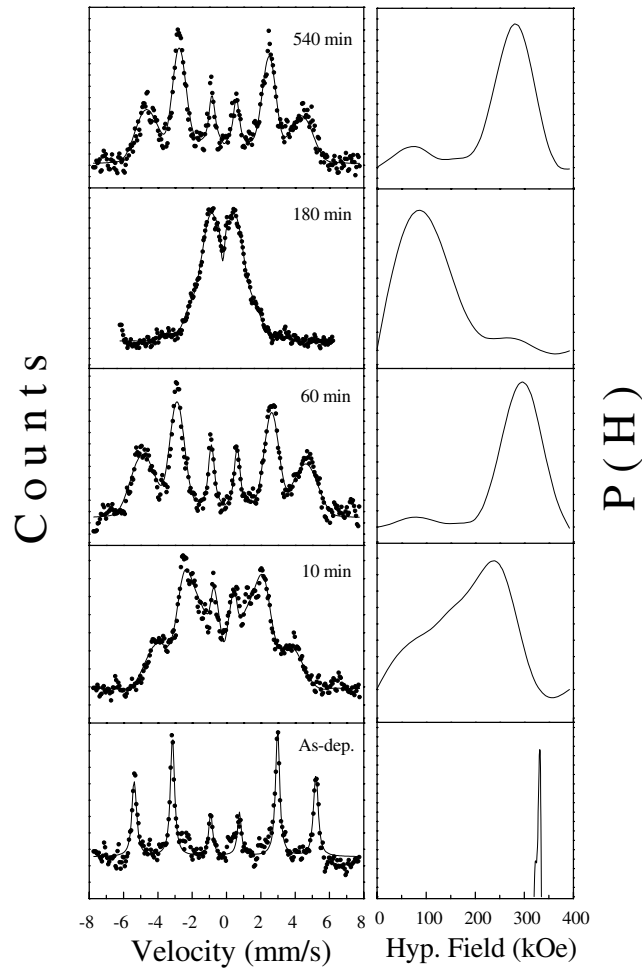


Figure 2. Magnetization obtained from the alternating gradient magnetometer.

( $\lambda = 1.5406 \text{ \AA}$ ) in an X-Pert Philips diffractometer. A scan step of 0.1 in the  $2\theta$  range from  $36^\circ$  to  $50^\circ$  was used with a fixed counting time of 60 s, and XAS in two regions—the near-edge structure (XANES) and the extended fine structure (EXAFS)—were obtained at Laboratório Nacional de Luz Síncrotron, Campinas, SP, Brazil. Room temperature CEMS measurements were performed using a constant-acceleration electromechanical drive system with a multichannel analyser for collecting and storing the data, equipped with a He- $\text{CH}_4$  proportional counter.  $^{57}\text{Co}$  in rhodium at room temperature was used as a source (25 mCi). The hyperfine parameters for the spectra were obtained with a least-squares fitting procedure assuming Lorentzian line-shapes. The magnetization measurements were performed at room temperature using a conventional alternating gradient magnetometer (AGM) with the applied field always parallel to the film surface.

### 3. Results and discussion

Figure 1 shows the XRD patterns for the samples. The typical multilayer modulation in the as-deposited sample is observed; it corresponds to a spacing period (about  $47 \text{ \AA}$ ) greater than the nominal thickness of the layers. After 10 min of thermal treatment at  $610^\circ\text{C}$ , the multilayer structure disappears and only one peak remains that is indexed to a (110) bcc reflection due to the FeV solid solution; the same peak is noticed for 60 min of annealing. The (110) peak dislocation to the right confirms the  $\alpha$ -phase formation. The Fe atoms diffuse to V layers at high temperature [10], and at  $610^\circ\text{C}$  the XRD results confirm Fe-V interdiffusion. A clear change of structure occurs with the 180 min annealed sample; these peaks can be indexed to a tetragonal  $\sigma$ -phase. With 540 min of annealing time the structure goes back to a cubic phase. This is an unexpected result. It is possible to observe from the magnetic measurement results (figure 2) that the as-deposited sample has a ferromagnetic behaviour with a reduction of magnetization intensity under the  $610^\circ\text{C}$  treatment; the reduction is larger for a 180 min treatment. These results confirm the structural transition to a  $\sigma$ -phase in this sample, with a decrease of magnetic moment [11]. With a 540 min thermal annealing treatment, there is a



**Figure 3.** CEMS spectra for samples subjected to thermal treatment at 610 °C for several durations. The  $P(H)$  curves are shown to the right of the corresponding spectra.

cubic phase present with the same magnetization as the disordered 10 min treatment bcc phase. The CEMS spectra can be seen in figure 3. The Wivel–Mørup model [12] was used to fit all the spectra. The  $P(H)$  distribution is also shown for each spectrum in figure 3 on the right. For the as-deposited sample, the  $P(H)$  curve consists of a sharp peak centred on 330 kOe that is characteristic of  $\alpha$ -Fe. A small almost-zero-field contribution (not considered in the fitting) could be due the Fe moment reduction at the interface layer. For the 10 min spectrum we observe an average magnetic hyperfine field (HF) reduction due to the 50–50 FeV alloy. The HF increases for the 60 min spectrum. The CEMS spectra confirm the magnetization results, with a HF reduction for 180 min treatment. An increase in the HF is observed for 540 min treatment with a  $P(H)$  curve similar to that for the 60 min spectrum. This HF corresponds to a Fe moment of about  $1.9 \mu_B$  and is compatible with a disordered bcc bulk alloy,  $\text{Fe}_{0.8}\text{V}_{0.2}$  [11]. A good indication of the sample behaviour can be given by the XANES results (figure 4), where it is possible to observe only one modification for the Fe signal (figure 4(a)) in the 180 min sample and several modifications in the V signal (figure 4(b)) in comparison to the iron and

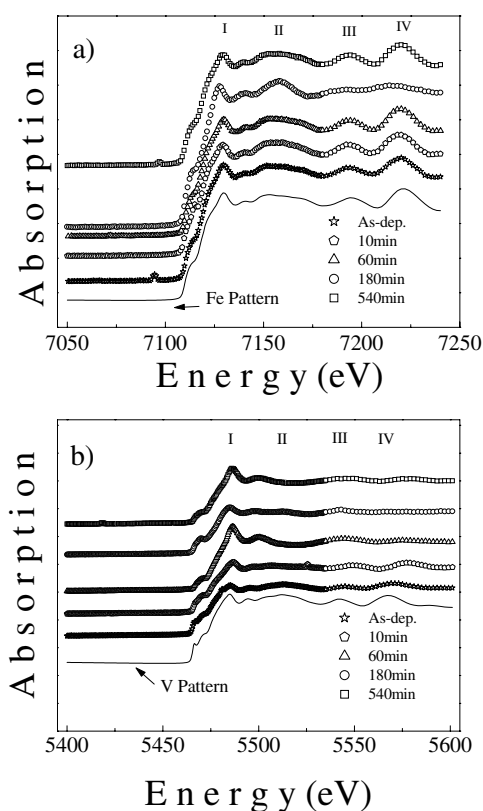
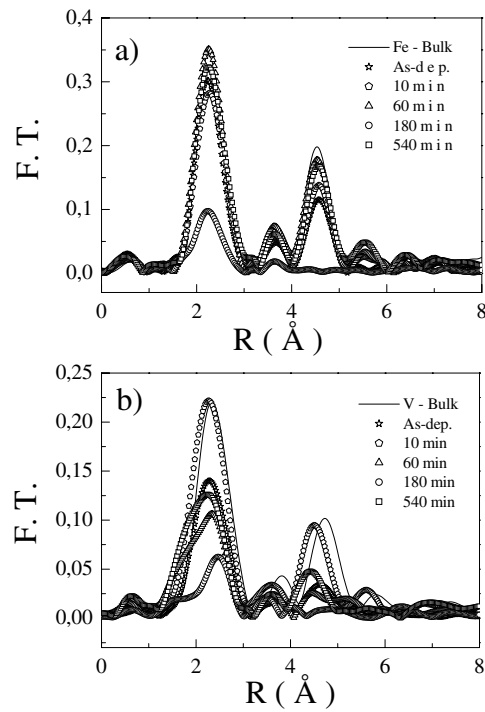


Figure 4. XANES near the Fe K edge (a) and near the V K edge (b).

vanadium references, respectively. The as-deposited, 10, 60, and 540 min iron signals have the same pattern as the iron reference with a bcc structure. The 180 min iron signal can be ascribed to a tetragonal structure on the basis of the modification in peaks II, III, and IV. The concavity in the second peak for the V signal in the 60 and 640 min samples can be correlated with a process of oxidation of vanadium, and this can occur with a migration of V atoms to the surface of the film, resulting in a vanadium-poor FeV solid solution, with possible back-diffusion of Fe [10].

The corresponding Fourier transform (FT) magnitude of EXAFS signals at the Fe K-edge is shown in figure 5(a). The iron reference is also shown for comparison. For the as-deposited sample the FT of the EXAFS spectrum has small amplitude as compared with that of the reference, while the local structure for the samples annealed for 10, 60, and 540 min is bcc but the magnitudes that represent the neighbouring configurations for each central atom (Fe) are different. The first peak is the contribution of nearest and next-nearest neighbours and the 60 min thermal treatment result is the closest to the iron reference result. It is possible to confirm this in the Mössbauer  $P(H)$  distribution. We observe a drastic reduction in the amplitude for 180 min annealing. This is compatible with a single  $\sigma$ -phase. For a 540 min thermal annealing the bcc  $\alpha$ -phase is restored with almost the same intensity as that of the standard iron. The V K-edge FT of the EXAFS (figure 5(b)) shows a more complex behaviour. The vanadium reference is also shown for comparison. It is possible to see that the 10 min annealed sample is the closest to the vanadium reference, with a reduction in the neighbour distances. The 180 min curve clearly displays a structural change that suggests that vanadium follows iron



**Figure 5.** Fe K-edge (a) and V K-edge (b) FT magnitudes of EXAFS signals for 610 °C annealed samples for several durations.

in the  $\alpha$ - $\sigma$ - $\alpha$  oscillation of the FeV thin film. For the 60 and 540 min samples, we observe superimposed on the first peak the oxygen contribution, as was observed in XANES results.

#### 4. Conclusions

The samples show a disorder–order–disorder transition in the FeV solid solution obtained after repeated thermal treatments at 610 °C of thin multilayers. The structural  $\alpha$ - $\sigma$ - $\alpha$  oscillation of the FeV solid solution for different thermal annealing times is the most significant aspect observed in this study and it is not reported in the literature available to the authors. A noticeable result is the retro-diffusion of V after 540 min of annealing, with a possible back-diffusion of Fe. This behaviour may be related to a process of oxidation in the sample, the oxide coming from the substrate layer of SiO<sub>2</sub> or from the sample itself, where the oxide can be formed during the manipulation of the sample between deposition and the thermal treatment.

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