CHEMISTRY LETTERS, pp. 59 - 62, 1984.

SYNTHESIS OF Fe-Mg MULTILAYERED FILMS WITH ARTIFICIAL SUPERSTRUCTURES

Teruya SHINJO,* Kenji KAWAGUCHI, Ryoichi YAMAMOTO,
Nobuyoshi HOSOITO, and Toshio TAKADA
Institute for Chemical Research, Kyoto University, Uji, Kyoto 611

By a vacuum deposition method, multilayered films with artificial superstructure have been prepared from mutually insoluble elements, Fe and Mg. X-Ray diffraction confirmed the establishment of very short artificial periods, e.g. Fe(0.8 nm)-Mg(2.4 nm). The multilayer deposition is a very promising technique to fabricate new materials.

The mutual solubility of Fe and Mg is much less than 1% even in the liquid state and no intermetallic compound exists in the solid state phase diagram. By ordinary metallurgical techniques Fe-Mg alloys can not be prepared and consequently there has been no report on the concentrated Fe-Mg alloys except a very recent work on amorphous alloys.[1)]
In this letter we report the preparation of multilayered films consisting of extremely thin Fe and Mg layers with artificial regularities.

The samples were prepared by alternate deposition in ultrahigh vacuum.
The main part of the deposition system is schematically illustrated in Fig. 1.
Two electron beam guns could heat Fe and Mg sources independently and the thickness of each layer was controlled by operating the two shutters over Fe and Mg hearths.
An oscillating quartz sensor was located near

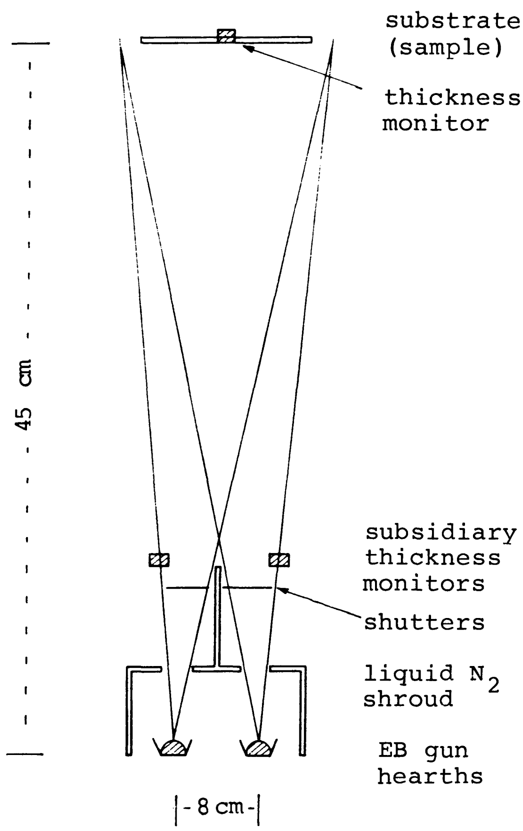

Fig. 1. The deposition system.

the substrate and connected to a programmable thickness monitor (Inficon IC6000), which controlled the shutter motions. The subsidiary thickness sensors near the shutters always checked the evaporation rates, independently of the shutter operation. With a cryopump (CTI Cryo10) the base vacuum attained to the 10^{-10} Torr (1 Torr = 133 Pa) range and the vacuum during the deposition process was kept in the 10^{-9} Torr range. The substrates (glass plates for X-ray and mylar films for Mössbauer measurements respectively) were cooled down to about -50 °C to inhibit the three dimensional grain growth. The combinations of Fe and Mg layer thicknesses in the prepared samples were as follows; (1)Fe(3 nm)-Mg(6 nm), (2)Fe(1.5 nm)-Mg(3 nm), (3)Fe(0.8 nm)-Mg(2.4 nm), (4)Fe(0.8 nm)-Mg(0.8 nm) and (5)Fe(0.4 nm)-Mg(0.4 nm). In each case, the deposition process was repeated for about 100 times.

If the films have periodic structures resulting from the alternate deposition of Fe and Mg, X-ray diffraction peaks due to the artificial periodicities should be observed. Figure 2 shows the X-ray diffraction pattern in a small angle region for the sample of Fe(0.8 nm)-Mg(2.4 nm). The 1st order Bragg peak is very sharp and strong. Higher order peaks (up to 5th) also are easily observed, which indicate the compositional modulation has a square wave mode. The length of artificial period is calculated from the observed peak angle using the Bragg formula. The obtained values for the prepared samples are shown in Fig. 3, with

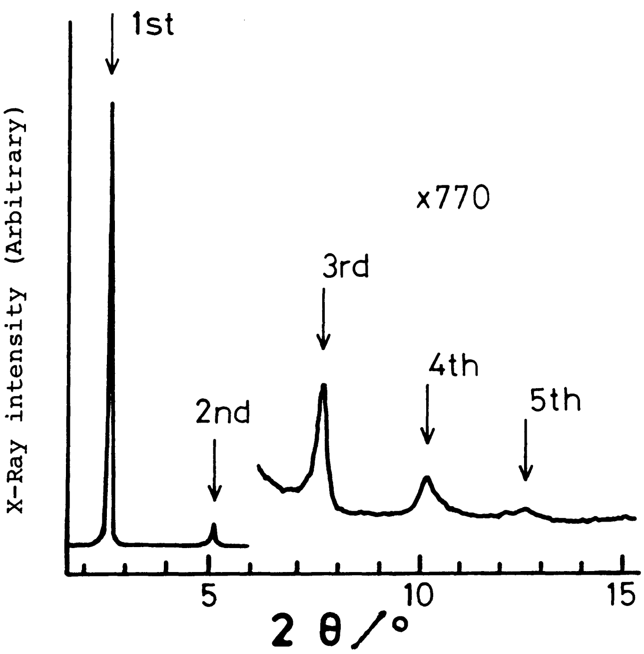

Fig. 2. X-Ray diffraction pattern for the film of Fe(0.8 nm)-Mg(2.4 nm).

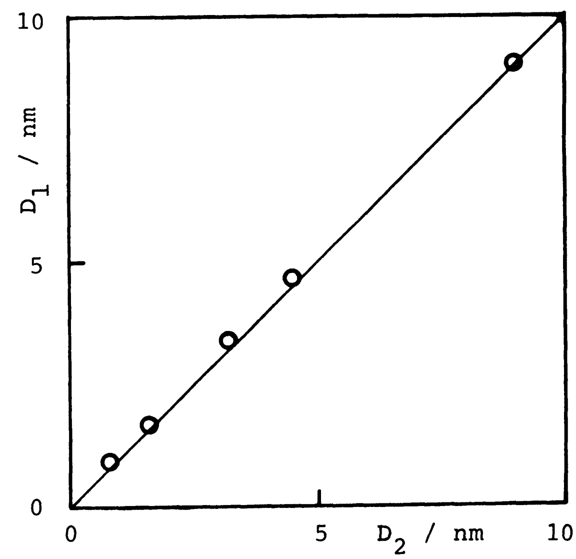

Fig. 3. Comparison of the artificial periods estimated from X-ray diffraction angle(D_1) and from the thickness monitor (D_2).

comparing the values determined by the thickness monitor (the sum of single Fe and Mg layer thicknesses). The agreement is excellent. Namely the planned periodicities have been exactly achieved in the deposited films. In the direction perpendicular to the film plane, the structure of the deposited film is controlled in an atomic scale.

In order to study the magnetic properties of Fe-Mg multilayers, ^{57}Fe Mössbauer measurements were carried out. For example, the result on the sample of Fe(0.8 nm)--Mg(2.4 nm) are reproduced in Fig.4. The Curie temperature decreases with the decrease of the Fe layer thickness and accordingly the spectrum at 300 K has no magnetic hyperfine structure. On the other hand, the spectrum at 4.2 K shows a six-line pattern, suggesting the Fe layers are entirely ferromagnetic. Although the line widths are fairly broadened, the average hyperfine field is near the standard α-Fe value and the range of the distribution is not very wide. In comparison with the results on Fe interfaces contacting with other elements,[2] it may be concluded that the electronic structure of interface Fe atoms in contact with Mg is not drastically modified. The study on the magnetic properties of Fe-Mg multilayer films combining the measurements with SQUID magnetometer is in progress and the results will be published elsewhere.

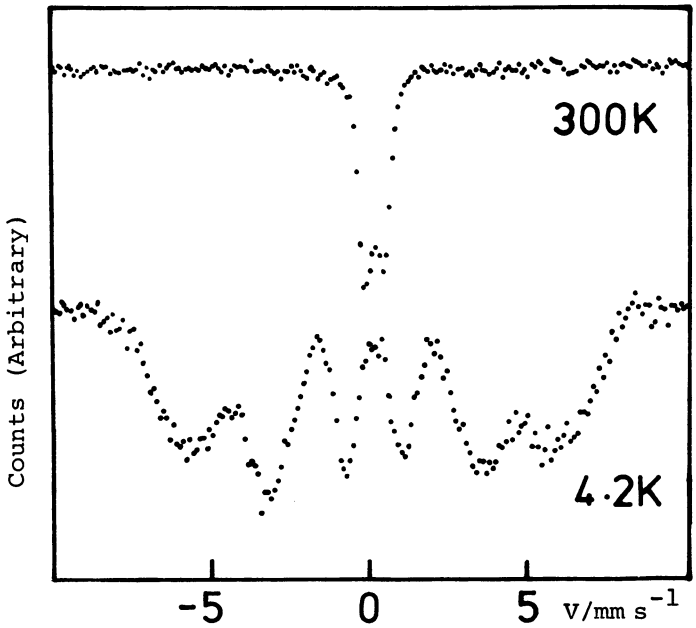

Fig. 4. Mössbauer absorption spectra at 300 K and 4.2 K for the sample of Fe(0.8 nm)-Mg(2.4 nm).

The preparation of Fe-Mg multilayered films was attempted many years ago by Dinklage for the purpose to fabricate a synthetic X-ray monochrometer.[3] However, he has only made X-ray measurements. No attention has been paid from a viewpoint of new material synthesis and there has been no report concerning the physical properties of Fe-Mg multilayered films. We have succeeded in preparing Fe-Mg multilayered films with much shorter artificial periods, of excellent qualities as were evidenced by the sharp X-ray diffraction peaks.

Preparation of amorphous alloys is another method to synthesize concentrated alloys in non-equilibrium states. By simultaneous evaporation, van der Kraan and Buschow have prepared Fe-Mg amorphous alloys.[1] In contrast to amorphous alloys, the multilayered films show sharp X-ray diffraction peaks. Therefore the uniformity can be checked microscopically and the crystallographic structure analysis is possible. The atomic arrangement in the multilayers can be artificially controlled to much higher degree than that of amorphous alloys.

It has been generally accepted that multilayered films with artificial superstructures can only be composed from very similar constituents such as Cu-Ni[4] or GaAs-AlAs.[5] The present result evidences that even from mutually insoluble elements, artificial superstructures with very short wavelengths are achieved. The difference of the atomic radii of Fe and Mg reaches to nearly 30%. Hence it is strongly suggested that the multilayer deposition technique is very promising to fabricate new materials which do not exist in the thermal equilibrium states.

This work was supported by the Grand-in-Aid for Special Project Research from the Ministry of Education, Science and Culture.

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(Received October 3, 1983)