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Amorphization in Fe-Si multilayers by solid-state reaction

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Solid-state reaction in Fe-Si multilayers occurring at 573 and 658 K has been studied. Iron is the predominantly diffusing species at lower temperatures. It is found that a large asymmetry in the mobilities of the two constituents is a basic requirement for amorphization to occur. A detailed study of transformation kinetics suggests that amorphization occurs via one-dimensional diffusion-controlled growth. Information about the possible mechanism of amorphization has been obtained.

I. INTRODUCTION

Recent observations on the amorphization of crystalline phases occurring at a constant temperature[1,2] have demonstrated that the amorphous state should be considered as one of the thermodynamically metastable states of the system, and not simply as a supercooled liquid state which would crystallize when there is a diffusion of its constituents. Crystal-to-glass transformation may occur either via a solid-state reaction between two components present in the form of thin multilayers, or as a result of hydrogen charging of an alloy phase. In the former case a high interface energy between different layers, and in the latter case the energy associated with lattice strains, give rise to a crystalline state with free energy higher than that of the metastable amorphous phase at the same temperature, thus providing the necessary driving force for the transformation. Amorphization in the solid state is generally observed in systems in which only one of the components has large diffusivity; it has been suggested that, while the mobility of only one species is sufficient to grow the metastable amorphous phase, mobility of both the components is necessary to bring in the atomic rearrangements necessary for nucleation and growth of crystalline phases.[2,3] Thus, if mobilities of the two constituents are highly asymmetric, then a temperature range can be found in which it is possible for the amorphous phase to grow while the formation of stable crystalline phases is still constrained. More recently, it has been suggested that[4] a large atomic-size mismatch between the two constituents may be the main criterion for the solid-state amorphization reaction, since in the system which exhibits amorphization by solid-state reaction, the ratio of the atomic volumes has been found to be $V_{\text{small}}/V_{\text{large}} < 0.6$.[5] It has been argued that in Zr/Co and Zr/Ni couples[6,7] growth of the amorphous layer proceeds from each interface of the unreacted components, thus consuming the Co(Ni) layer. Therefore, the higher mobility of the smaller constituent atom into the larger one may be a more dubious necessity. Thus, ambiguities exist regarding the basic requirements for solid-state-reaction-induced amorphization to occur. Fe/Si is a system in which the possibility of solid-state amorphization has been documented.[8,9] The system has a negative heat of mixing, and diffusivity of the constituents is high even at temperatures well below the formation temperature of the first intermetallic compound.[10] Therefore, solid-state reaction around 573 K results predominantly in the formation of the amorphous phase, while annealing at higher temperatures results in crystalline intermetallic compounds.[9] In the present work we report the results of a detailed study of amorphization by solid-state diffusion in Fe/Si multilayers. Mössbauer spectroscopy in conjunction with other complementary analytical techniques has been used to get information about the formation of amorphous and crystalline phases and the kinetics of the transformation process. As the Fe/Si system has been extensively studied by Mössbauer spectroscopy and the hyperfine parameters of various intermetallic compounds, solid solution as well as amorphous

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phases, are known, the technique can give precise information about the formation of various phases in the system.

II. EXPERIMENTAL

Multilayers of Fe/Si, consisting of three layers each of Fe and Si, were deposited on a Si/SiO$_2$ substrate by successive evaporation of the two elements in a vacuum of 6×10^{-5} Pa. The deposition rate was kept at 0.2–0.4 nm/s. The substrate was kept at room temperature during the evaporation. Relative thicknesses of Fe and Si layers were chosen so as to achieve an overall composition of Fe$_{50}$Si$_{50}$. Two sets of specimens were prepared with the following sequence of layers: set A, substrate/Fe (12.4 nm)/Si (21.0 nm)/^{57}Fe (6.2 nm)/Fe (6.2 nm)/Si (21.0 nm)/Fe (12.4 nm)/Si (21.0 nm), and set B, substrate/Fe (8.0 nm)/Si (13.6 nm)/Fe (2.0 nm)/^{57}Fe (4.0 nm)/Fe(2.0 nm)/Si (13.6 nm)/Fe (8.0 nm)/Si (13.6 nm) (Fig. 1). The thin ^{57}Fe marker layer either interfaced with Si (set A) or placed in the center of an Fe layer (set B) makes it possible to look selectively at the Fe/Si interface or away from it using Mössbauer spectroscopy. Auger electron depth profiles of the as-deposited specimens *A* and *B* (Fig. 2) indicate rather sharp interfaces between Fe and Si layers. Also, the oxygen and carbon contamination of the specimens was found to be quite low.

Solid-state reaction was induced in the specimens by isothermal annealing at 573 or 658 K in an atmosphere of flowing nitrogen gas. Secondary-ion mass spectrometry (SIMS) depth profiles were obtained with a CAMECA ims-4f ion microscope by 5.5-keV cesium bombardment and by positive-ion detection. ^{161}CsSi$^+$, ^{189}CsFe$^+$, and ^{190}CsFe$^+$ molecular ions were used to measure the silicon, iron, and ^{57}Fe profiles, respectively. This choice was due to the linear behavior of the CsM^+ ion yield, which makes the analysis independent of the matrix effects.[11] X-ray diffraction (XRD) measurements (Co $K\alpha$ radiation) were done with a conventional θ-2θ goniometer in the Bragg-Brentano parafocusing geometry. After a preliminary analysis in the 2θ range of 20°–120°, the spectra were collected in the 29–36 nm^{-1} reciprocal space range encompassing the Fe(110) and FeSi(211) peaks. Conversion-electron Mössbauer spectra were measured in constant acceleration mode, with a 50-mCi ^{57}Co:Rh source and a gas-flow (95% He, 5% CH$_4$) proportional counter. The spectra were computer fitted to determine the distribution of hyperfine parameters using a fitting program developed by Le Caer and Dubois.[12]

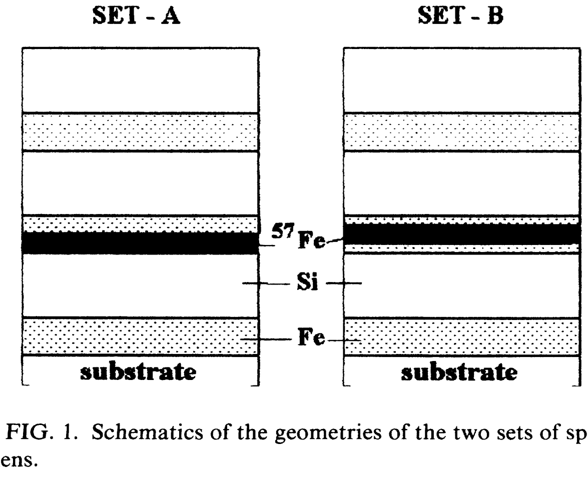

FIG. 1. Schematics of the geometries of the two sets of specimens.

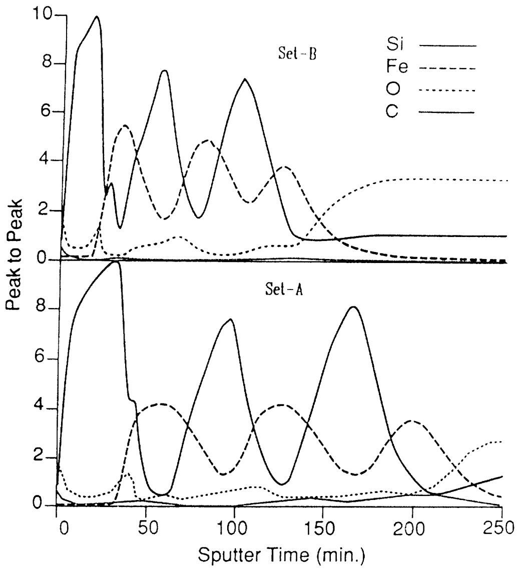

FIG. 2. Auger electron depth profile of the as-deposited specimens *A* and *B*.

III. RESULTS AND DISCUSSION

Figure 3 gives the SIMS profiles of the specimen *A* in the as-deposited state as well as after annealing at 573 K for 57 h and at 658 K for 4 h. Although it is difficult to get quantitative information about the depth distribution of different constituents from SIMS, qualitatively one may note that, after annealing at 573 K for 57 h, the depth profiles of ^{56}Fe and ^{57}Fe have considerably broadened while the width of the Si profile is not significantly affected. This observation suggests that Fe is the faster diffusing species in this system, the diffusivity of Si being relatively insignificant at 573 K. On the basis of the ratio of the atomic radii of the constituents also, one expects the diffusivity of Fe to be higher. On the other hand, annealing at 658 K for 4 h results in significant broadening of both Si and Fe depth profiles, indicating that at this temperature the diffusivity of Si has become comparable to that of Fe. In an earlier study on solid-state reaction in the Fe-Si system,[10] Lau *et al.* have unambiguously concluded, using a thin tungsten marker, that at temperatures of 673 K and above, silicon is the main diffusing species. However, in these experiments a thin layer of iron was deposited upon a single crystalline wafer of Si, the deposition rate being an order of magnitude higher than that in the present case. Therefore, a higher concentration of defects in the iron layer as compared to the silicon and a higher annealing temperature may be the cause of the enhanced silicon diffusivity in iron in their specimen. In order to obtain a rough esti-

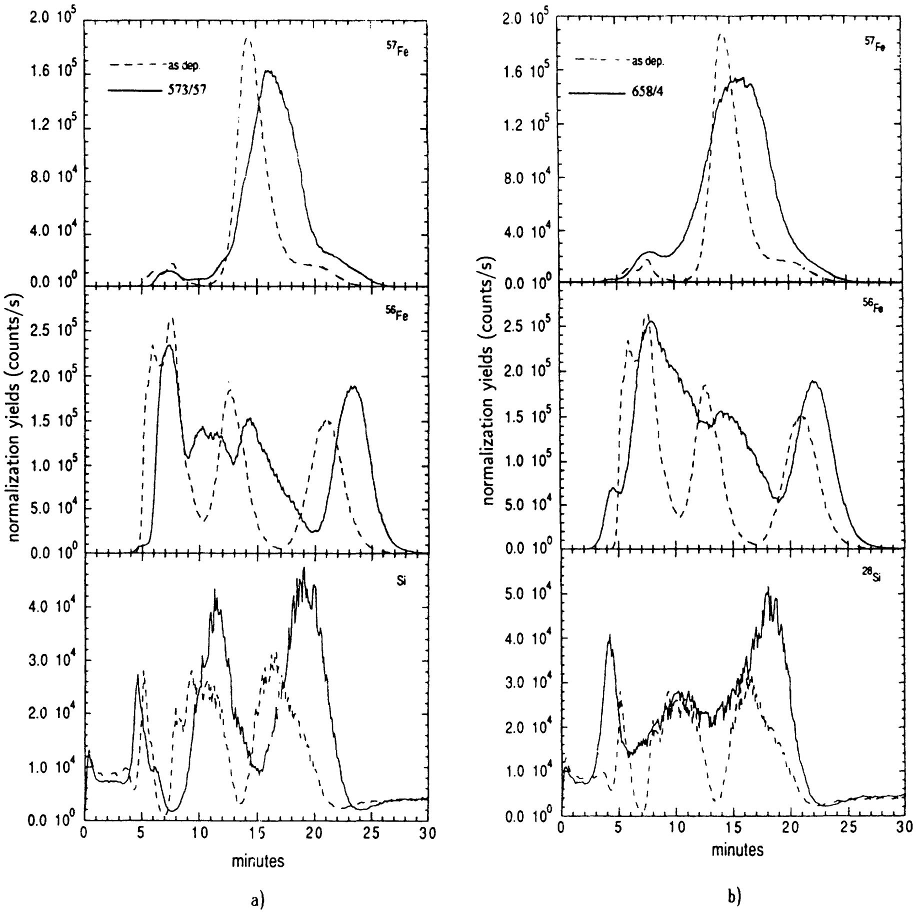

FIG. 3. Comparison of the SIMS depth profiles of various isotopes in specimen *A* in the as-deposited state with those in (a) specimen *A* annealed at 573 K for 57 h, and (b) specimen *A* annealed at 658 K for 4 h.

mate of the diffusivity of iron in Si at 573 K from the SIMS data, one may assume that the initial depth profile of ^{57}Fe is a Gaussian. The time scale may be calibrated in terms of sputtered depth by taking the width of the ^{57}Fe depth profile in the as-deposited specimen to be 6 nm. The atomic diffusion at 573 K will result in a broadening of the profile, and the diffusion coefficient can be obtained using the formula[13]

$$D=(\Omega_2^2-\Omega_1^2)/2t\ , \tag{1}$$

where Ω_1 and Ω_2 are the standard deviations of the depth profiles before and after diffusion and t is the annealing time. From the observed values of 3 and 5.9 nm for Ω_1 and Ω_2, respectively, one obtains an estimate of the diffusivity of iron in silicon at 573 K to be 6.1×10^{-19} $\mathrm{cm^2\,s^{-1}}$. Figure 4 gives the XRD pattern of specimen *A* after different stages of annealing. Annealing at 573 K for 57 h causes the position of the Fe(110) peak to shift to lower *d* values. This may be attributed to some structural relaxation or to the dissolution of Si in the iron layers, causing the lattice to contract. No formation of any additional phases could be detected. After the annealing at 658 K for 4 h, the appearance of an additional peak around $d=0.183$ nm, corresponding to the FeSi(211) reflection, provides evidence for the formation of the FeSi phase. It may be noted that the position of the FeSi(210) reflection is very close to the Fe(110) reflection (a few hundredths of nm^{-1} lower). Therefore, formation of the FeSi phase would also contribute to an apparent shift in the position of the Fe(110) reflection. Figure 5 gives the Mössbauer spectra of the specimen *A* annealed at different temperatures. The zero of the velocity scale corresponds to the centroid of the Mössbauer spectrum of α-Fe at 300 K. The spectrum of the as-prepared speci-

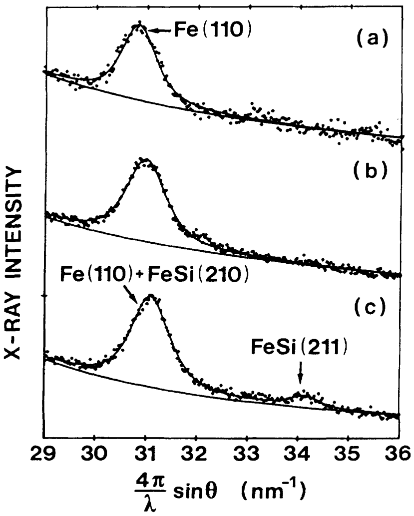

FIG. 4. X-ray diffraction pattern of specimen *A* (a) in the as-deposited state, (b) after annealing at 573 K for 57 h, and (c) after annealing at 658 K for 4h.

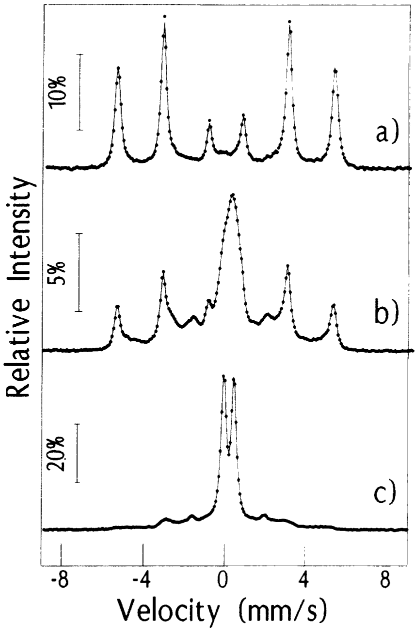

FIG. 5. Conversion electron Mössbauer spectra of specimen *A* (a) in as-deposited state, (b) after annealing at 573 K for 57 h, and (c) after annealing at 658 K for 4h.

men consists predominantly of a sharp sextet with hyperfine magnetic field $B_{\rm hf}=33.3$ T, which corresponds to α-iron. A small amount of low-field components suggests that, in agreement with the results of Auger electron depth profiling, the interfaces between different layers are rather sharp. A detailed analysis of the spectrum of the specimen annealed at 573 K for 32 h, as described in the following paragraphs, suggests that it consists of three components: a sharp sextet around 33.0 T, a broad magnetic component, and a broad paramagnetic doublet. While the sharp sextet corresponds to α-Fe, the shape of the paramagnetic component plus a part of the broad magnetic component resemble very well the Mössbauer spectrum of an amorphous Fe-Si phase.[14] Since the XRD of this specimen does not indicate formation of any new crystalline phase, while from the Mössbauer spectrum one finds that about 60% of ^{57}Fe goes from α-Fe to the new phase, one can conclude that the new phase formed as a result of annealing at 573 K is predominantly an amorphous phase. The spectrum of the specimen annealed at 658 K for 4 h consists predominantly of a sharp doublet with its hyperfine parameters (isomer shift $\delta_{\rm IS}=0.20$ mm s^{-1}, quadrupole splitting $\Delta_{\rm QS}=0.50$ mm s^{-1}) corresponding to the crystalline FeSi intermetallic compound.[15] Thus, in conformity with the result of XRD measurements, Mössbauer spectroscopy too evidences the formation of the crystalline FeSi phase after annealing at 658 K. The absence of any sextet corresponding to α-Fe suggests that the whole of the 6.2-nm-thick ^{57}Fe layer has undergone reaction with the Si layer.

Thus, the results of Mössbauer measurements combined with those of XRD measurements show that, while solid-state reaction at 573 K results predominantly in the formation of an amorphous phase, solid-state reaction at 658 K causes formation of crystalline FeSi. At the same time, from SIMS measurements one finds that, while at 573 K there is a large difference in the mobilities of Fe and Si, the two become comparable at 658 K. Thus, a large asymmetry in the mobilities of the two constituents appears to be a basic requirement for the solid-state amorphization. A large atomic-volume mismatch ($V_{\rm small}/V_{\rm large}<0.6$),[4] observed in the systems amorphized so far by solid-state reaction, may be the origin of this difference in the mobilities of the two constituents. Figures 6 and 7 give the Mössbauer spectra of specimens *A* and *B* in as-prepared condition as well as after isothermal annealing at 573 K for various periods of time. With increasing annealing time a broad paramagnetic doublet as well as a broad sextet grow in intensity at the expense of the α-iron sextet, indicating progressive stages of solid-state reaction. The spectra were computer fitted to obtain two distributions; a distribution of quadrupole interaction corresponding to the paramagnetic component and a distribution of the hyperfine magnetic fields to account for the broad magnetic component. A linear

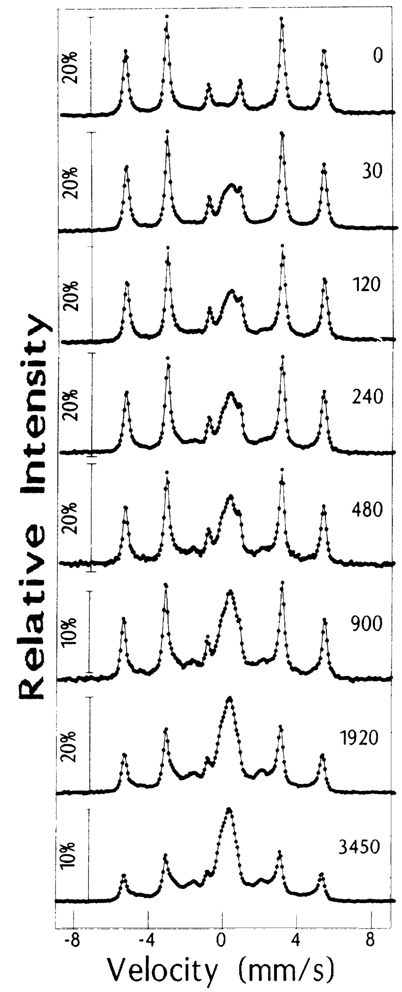

FIG. 6. Conversion electron Mössbauer spectra of specimen *A* annealed at 573 K for different periods of time (given in minutes).

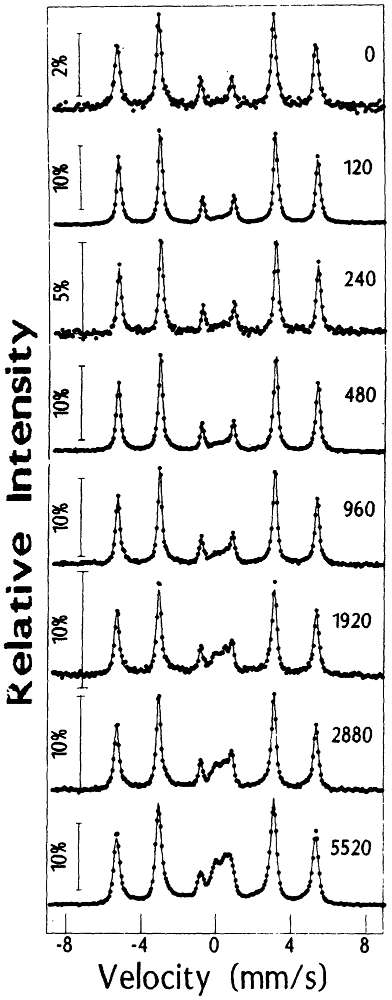

FIG. 7. Conversion electron Mössbauer spectra of specimen *B* annealed at 573 K for different periods of time (given in minutes).

correlation between δ_{IS} and B_{hf} of the form $\delta_{IS}=\alpha+\beta B_{hf}$ was assumed for the magnetic hyperfine field distribution. The values of α and β determined by minimizing the χ^2 were 0.45 $mm\,s^{-1}$ and -0.0138 $mm\,s^{-1}T^{-1}$, respectively. These values of the correlation parameters agree very well with the known variation of δ_{IS} with B_{hf} in solid solutions and intermetallic compounds of Fe-Si.[16,17] For the quadrupole distribution also, a linear correlation between δ_{IS} and Δ_{QS} of the form $\delta_{IS}=0.28-0.033\Delta_{QS}$ (both δ_{IS} and Δ_{QS} measured in mm/s) was taken in order to account for the asymmetry of the paramagnetic doublet. In annealed specimens it was found necessary to introduce an additional doublet with $\delta_{IS}=0.05$ $mm\,s^{-1}$, $\Delta_{QS}=0.33$ $mm\,s^{-1}$, $\Gamma=0.27$ $mm\,s^{-1}$, which noticeably improved the χ^2. The parameters of this doublet approximately correspond to those of $FeSi_2$. The resultant distributions of quadrupole interactions as well as hyperfine field in specimen *A* are shown in Fig. 8. In order to determine the amount of unreacted α-Fe, the spectra were refitted by taking a separate sharp sextet corresponding to the α-Fe phase, in addition to the distributions of quadrupole interaction and magnetic hyperfine fields. Table I gives the relative areas under various components as a function of annealing time.

The broad doublet in the spectra of Figs. 6 and 7 cannot be identified with any crystalline intermetallic compound or solid solution of Fe-Si. On the other hand, studies of sputter-deposited amorphous Fe-Si alloys have shown that for 50 at. % or more Si concentration the Mössbauer spectrum consists of a broad doublet only, while for lower Si concentration it is an overlap of a doublet and a magnetic sextet.[14] Thus, the broad doublet and part of the magnetic component observed in the annealed specimens can be attributed to the amorphous phase. As discussed earlier, the fact that, even after annealing at 573 K for 57 h the XRD does not show formation of any new crystalline phase, also provides evidence that the new phase formed as a result of annealing (as evidenced by Mössbauer spectroscopy) must be amorphous in nature. Because of the overlapping contributions of the amorphous phase and Fe-Si solid solution to the mag-

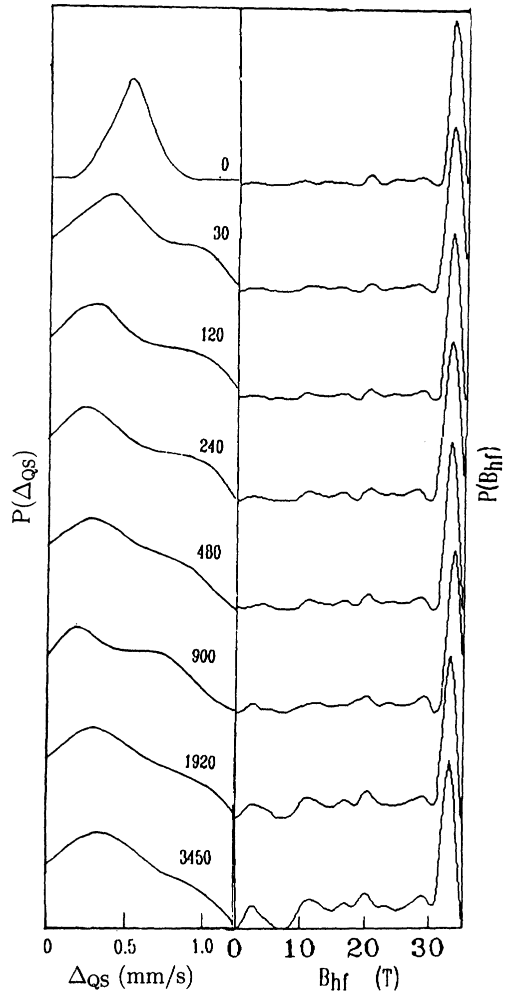

FIG. 8. Distribution of quadrupole interactions and hyperfine fields of specimen *A* after different times of annealing (in minutes) at 573 K. Several broad peaks in the hyperfine field distribution roughly correspond to hyperfine fields of Fe-Si solid solution and the known compounds of Fe-Si.

TABLE I. Relative areas of various Mössbauer components in specimen A after annealing at 573 K for different periods of time.

Annealing time (min)	Percentage area of α-Fe	broad magnetic component	nonmagnetic component
0	77.7	13.8	8.5
30	57.7	12.8	29.5
120	54.5	15.8	29.7
240	49.3	18.3	32.4
480	44.8	20.9	34.3
900	37.9	24.0	38.1
1920	24.7	32.1	43.2
3450	18.6	34.1	47.3

netic hyperfine field distribution, it is not possible to separate the two and thus to determine their relative amounts. Further, the quadrupole splitting of the intermetallic compound FeSi ($\Delta_{QS}=0.527$ mm s^{-1})[15] lies within the range of the Δ_{QS} distribution of the amorphous phase, and thus a possible presence of FeSi in the specimen cannot be ruled out. However, the absence of any sharp peak in the quadrupole splitting distribution around 0.527 mm s^{-1} suggests that the amount of FeSi phase, if present, should be small. The quadrupole doublet with $\delta_{IS}=0.05$ mm s^{-1} and $\Delta_{QS}=0.33$ mm s^{-1} may be attributed to the FeSi$_2$ phase. However, the possibility of some oxide phase of Fe-Si with these Mössbauer parameters cannot be ruled out.

From Fig. 8 it can be noted that in all the spectra the hyperfine field distribution contains, besides the α-Fe peak around 33 T, several broad peaks which roughly correspond to the known hyperfine components of bcc Fe-Si solid solution (30.9 and 28.2 T),[16] and Fe$_3$Si (31 and 20.1 T).[17] The lower field components may be attributed to an amorphous Fe-Si phase with Fe concentration greater than 50 at. %.[14]

The above analysis, based on both XRD and Mössbauer results, suggests that an annealed specimen contains predominantly three phases: (i) pure α-iron, (ii) a bcc Fe-Si solid solution, and (iii) an amorphous Fe-Si phase. Small amounts of FeSi, FeSi$_2$, and FeSi$_3$ intermetallic compounds may also be present. It may be noted that the widths of both quadrupole splitting and magnetic hyperfine field distributions are much higher compared to those of amorphous Fe-Si films of various compositions formed by codeposition.[14,15] Since the positions of the peaks in quadrupole splitting as well as magnetic hyperfine field distributions of amorphous Fe-Si show a shift with the composition,[14] the broader distributions in the present case may be attributed to a composition gradient in the amorphous phase. Perusal of Table I shows that after annealing for about 50 h, while the relative amounts of nonmagnetic and broad magnetic components in specimen B are almost equal, in specimen A, the amount of nonmagnetic component is substantially higher than that of the broad magnetic component. Since in specimen B Mössbauer spectroscopy selectively looks at the region away from the Fe-Si interface, the above observation suggests that, as expected for a one-dimensional diffusion-controlled growth, a concentration gradient exists in a direction normal to the Fe-Si interface and the region away from the interface (toward the Fe layer) is richer in Fe.

Following Deal and Grove,[18] for large reaction times, the thickness d of the reacted interlayer is related to the reaction time by

$$d=-A/2+B^{1/2}t^{1/2}\ , \tag{2}$$

where B is the diffusion coefficient and B/A gives the interface velocity at the reaction front. Thus, the plot of X vs $t^{1/2}$ can yield the diffusivity of Fe in the present case. The thickness of the reacted Fe layer at either of the interfaces of a given Fe layer can be determined using the relations

$$d=X(t)\frac{D}{2}\quad \text{for set } A \tag{3}$$

and

$$d=0.5[0.978+1.022X(t)]\frac{D}{2},\quad \text{for set } B\ , \tag{4}$$

where $X(t)$ is the fraction of α-Fe reacted with Si after annealing for time t, and D is the thickness of a single iron layer. In deriving Eq. (4), 2.2% ^{57}Fe present in the natural iron layer has also been taken into account. In Fig. 9 is plotted the thickness of reacted α-Fe as a function of the square root of the annealing time at 573 K for specimen A. One may note that in the first 30 min the transformation occurs at a much faster rate. For longer annealing times the transformation follows approximately the shifted $t^{1/2}$ law predicted by the model of Deal and Grove. The initial faster reaction rate may be attributed to a higher disorder in the interface region. It may be noted that, even in the as-evaporated specimen, about 20% of Fe atoms lie in the interface region (Table I). A faster interdiffusion in the disordered region would cause the initial reaction rate to be faster. A second factor which can contribute to the initial higher slope of the transformation curve is the fact that, before the start of formation of the amorphous layer, the reaction rate is

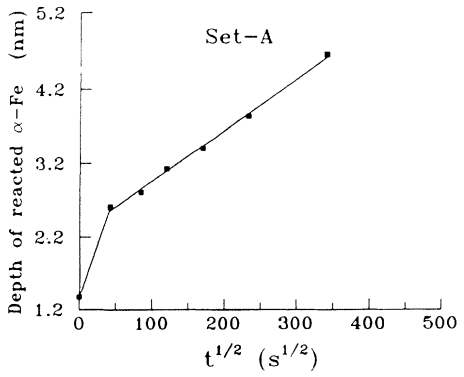

FIG. 9. Depth of reacted α-iron layer as a function of the square root of the annealing time at 573 K for specimen A.

determined by the diffusivity of Fe in Si. However, once an amorphous layer is formed, further dissolution of Fe in Si will be governed by the diffusivity of Fe through the intermediate amorphous layer, which is expected to be much lower than the diffusivity of Fe in Si.[19] The slope of the second part of the curve essentially represents the diffusion of Fe in the amorphous layer. The diffusivity of Fe in the amorphous layer at 573 K, determined by least-squares fitting the second part of the curve with a straight line, comes out to be $D(573)=4.77\times10^{-19}$ $\mathrm{cm^2\,s^{-1}}$ and is in reasonable agreement with the estimate obtained from SIMS measurements. In a similar way, the diffusivity of Fe obtained from the data of specimen *B* comes out to be 4.67×10^{-20} $\mathrm{cm^2\,s^{-1}}$. The two values differ considerably from each other. Further, the value of diffusivity estimated by Yan *et al.*[8] in a similar system at 573 K was found to be 6.0×10^{-17} $\mathrm{cm^2\,s^{-1}}$. This variation in diffusivity from specimen to specimen may be attributed to different states of the films; internal strains and defects in the films may depend upon the nature and temperature of the substrate, thickness of individual layers, deposition rate, etc., which in turn would affect the diffusivities of the constituents. From Fig. 9 one may note that a noticeable decrease in the reaction rate occurs after long annealing times ($t>32$ h). This may be attributed to the relaxation of internal stresses present in the as-evaporated multilayers on annealing, as evidenced by the XRD measurements also. Earlier studies have shown that the stresses in the crystalline layers influence the reaction rate.[2,20] Annealing of the specimen causes internal stress to relax, making diffusivity vary with annealing time, and thus resulting in an annealing-time dependence of the transformation rate.

Figure 10 gives the results of analysis of the transformation process on the basis of the Johnson-Mehl-Avrami formalism.[21,22] Assuming the reaction to follow Avrami kinetics, the fraction $X(t)$ of Fe reacted with Si can be written as

$$X(t)=1-\exp(-t/\tau)^n\ , \tag{5}$$

t being the annealing time, the time constant τ depending upon the activation energy of the process, and n being the Avrami exponent which depends upon the nature of the transformation process. Thus, the plot of $\ln\{-\ln[1-X(t)]\}$ versus $\ln(t)$ gives a straight line with its slope yielding the value of the Avrami exponent n. From Fig. 10, the value of n is obtained as 0.42 which is close to 0.5, a value characteristic of a one-dimensional diffusion-controlled growth.

In some recent XRD measurements on solid-state amorphization in the Ni-Hf system, the results have been interpreted in terms of a faster Ni diffusion along the grain boundaries in Hf layers.[23] In such a situation the dimensionality of the growth process would be more than 1 and consequently the Avrami exponent should have a value greater than 0.5. Therefore, the value of $n=0.42$ obtained in the present case provides evidence against iron diffusion along the grain boundaries in the Fe-Si system. A significant concentration gradient along a direction normal to the interface also suggests a one-dimensional diffusion-controlled growth. Thus, our analysis suggests that, at least in the present system, a substantial diffusion of Fe along the grain boundaries can be ruled out.

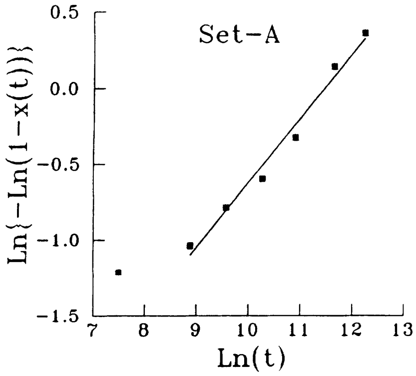

FIG. 10. Johnson-Mehl-Avrami plot for specimen *A*.

IV. CONCLUSIONS

Solid-state reaction in Fe-Si multilayers has been studied. Annealing at 573 K causes formation of an amorphous Fe-Si phase along with bcc Fe-Si solid solution, and possibly small quantities of Fe_3Si, FeSi, and $FeSi_2$ intermetallic compounds. Solid-state reaction at 658 K causes formation of crystalline FeSi. Iron is found to be the predominantly diffusing species at 573 K. It is found that a large asymmetry in the mobilities of the two constituents is a basic requirement for the amorphization to occur. The solid-state transformation follows a shifted $t^{1/2}$ law with interdiffusion coefficient exhibiting significant variation from specimen to specimen, which may be attributed to variations in the state of the films due to different preparation conditions and film thicknesses. Structural relaxation in the multilayers with annealing time causes the reaction rate to slow down with time. The present studies suggest that the amorphization occurs via a one-dimensional diffusion-controlled growth, and rule out any significant diffusion of Fe along grain boundaries in the Si layers.

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