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### ► To cite this version:

Hina Verma, Karine Le Guen, Renaud Delaunay, Iyas Ismail, Vita Ilakovac, et al.. Study of Buried Interfaces in Fe/Si Multilayer by hard X-ray emission spectroscopy. Surface and Interface Analysis, 2021, 53 (12), pp.1043-1047. 10.1002/sia.7005 . hal-03428877

**HAL Id: hal-03428877**

**<https://hal.sorbonne-universite.fr/hal-03428877>**

Submitted on 15 Nov 2021

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# Study of Buried Interfaces in Fe/Si Multilayer by hard X-ray emission spectroscopy

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## Abstract

Hard x-ray emission spectroscopy (XES) has been used to study buried layers and interfaces in a Fe/Si periodic multilayer. Until now, buried layers could be studied using the XES in the soft x-ray range. Here we extend the methodology to study the buried interfaces in hard x-ray region (photon energy  $\geq 5$  keV). We report the formation of FeSi<sub>2</sub> at all the interfaces with thicknesses of 1.4 nm. X-ray reflectivity measurements enable us to deduce the structure and thickness of the multilayer stack, thereby confirming the presence of FeSi<sub>2</sub>.

**KEYWORDS:** X-ray emission spectroscopy, Buried interfaces, Multilayer.

## 1. Introduction

The study of periodic multilayers is a topic of interest because they are used in optical components for numerous applications in x-ray mirrors, x-ray spectroscopy, space telescopes, and quantum well lasers. The optical performance of these multilayers dramatically depends on the interfaces formed between different layers of the multilayer stack. Hence, it is essential to characterize them and obtain important information regarding the multilayer structure, i.e. thickness, roughness, and density of the layers and their interfaces [1].

X-ray emission spectroscopy (XES) has proven to be a suited tool for analyzing buried layers and interfaces [2,3]. The emission spectra allow us to study the interactions between elements in the buried layers from the analysis of their valence states. The emission band obtained as a result of the valence to core transition, has a shape and width reflecting the chemical state of the emitting element. Hence, XES was shown to provide sensitive information about the physical chemical environment of the emitting elements in multilayers[4–7]. Until now XES has been applied in the soft x-ray region (photon energy  $\leq 2$  keV) to study the buried layers and interfaces.

The Fe/Si multilayer system has been of particular interest owing to its applications in microelectronics and magnetism [8]. The soft x-ray fluorescence measurements on the Fe/Si multilayer performed previously suggest that a strong interdiffusion between Fe and Si layers gives rise to a silicide layer at the interface [9]. Fe<sub>3</sub>Si, FeSi and FeSi<sub>2</sub> silicides have been reported to be formed at the interfaces depending upon the thickness and deposition conditions of the constituting Fe and Si layers. The iron rich, Fe<sub>3</sub>Si phase usually forms when the thickness of the Si layer is much lower than the thickness of the Fe layer, followed by the FeSi and FeSi<sub>2</sub> phases with the increase in Si layer thickness with respect to that of Fe [9,10]. The Si L<sub>2,3</sub> emission spectra for Fe/Si multilayer, studied by Imazono *et al.* [9], Carlisle *et al.* [11] and Watanabae *et al.* [12] reveal that intermixing takes place. The measured Si L emission spectra for the Fe/Si multilayer is possibly a mix of spectra of pure Si and other silicides. Similar results were obtained on the Mo/Si system, wherein the interfaces are composed of MoSi<sub>2</sub>, Mo<sub>5</sub>Si<sub>3</sub> [7,13]. The measured spectrum was decomposed with the reference spectra of the silicide compounds. From this decomposition, the interfacial thicknesses were deduced [12] as well as the chemical environment of the emitting element.

We want to extend this methodology to the hard x-ray range and evaluate the sensitivity of the technique in the same manner. We also performed X-ray reflectivity (XRR) measurements to

obtain preliminary structure of the multilayer stack, the thickness, density of the layers and possible interdiffusion between the layers.

## 2. Experimental Details

### 2.1 Sample Preparation

We prepared two Fe/Si multilayer samples. The first was made of 40 bilayers with alternating Fe and Si layers of 2.5 nm thickness each, and the second was made of 13 bilayers with alternating Fe and Si layers of thickness 3.6 nm and 4.5 nm respectively. We also prepared a reference sample of 20 nm thin film of Fe deposited on a Si substrate. Magnetron sputtering was used for the deposition at room temperature of samples on a Si (100) substrate using high purity Fe and Si targets. The argon pressure inside the chamber was maintained at  $1.9 \times 10^{-3}$  mbar during deposition. The base pressure was  $1.7 \times 10^{-7}$  mbar.

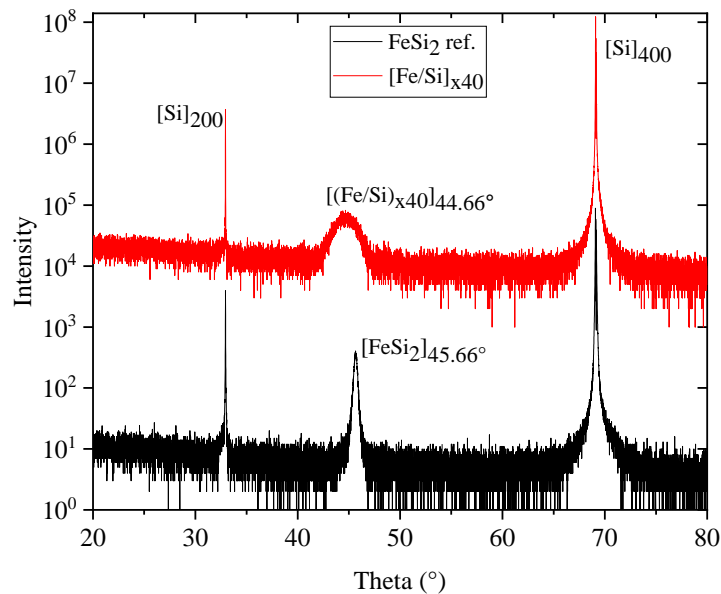
The periodic Fe/Si multilayer sample made of 40 bilayers was prepared at the deposition rate of 0.32 nm/s and 0.21 nm/s respectively with 150 W power set at each of the Fe and Si sputtering targets. The 20 nm thin film of Fe was fabricated with similar conditions. This Fe thin film sample is used as the Fe reference.

In the Fe/Si multilayer sample with 13 bilayers, the Fe and Si thicknesses were chosen so that the overall stoichiometry is  $\text{FeSi}_2$ . This multilayer was prepared at a deposition rate of 7.5 nm/s and 6.0 nm/s with the power of 150 W and 50 W maintained at the Fe and Si targets respectively. Various silicides,  $\text{FeSi}$ ,  $\text{FeSi}_2$  and  $\text{Fe}_3\text{Si}$  have been reported to be formed by the intermixing of Fe and Si layers at the Fe-on-Si and Si-on-Fe interfaces [11,14]. The  $\text{Fe}_3\text{Si}$  and  $\text{FeSi}$  silicides are found to be thermodynamically stable at annealing temperatures between 400-500°C [15]. However, at higher annealing temperatures (>600°C)  $\text{FeSi}_2$  forms at the expense of other silicides. It was observed that annealing temperatures between 600-700°C for 1-2 hours are sufficient to change 100 nm thick metal film deposited on a silicon substrate into a disilicide film [16–19]. From these observations, we deduce that when the  $[\text{Fe}(3.6\text{nm})/\text{Si}(4.5\text{nm})]_{\times 13}$  multilayer sample is annealed at 675°C for 2 hours under vacuum, rigorous interdiffusion and intermixing occur at interfaces throughout the multilayer stack. The stoichiometry of the Fe and Si layers along with the annealing conditions drives the formation of  $\text{FeSi}_2$  in the bulk. The presence of this  $\text{FeSi}_2$  was confirmed with the x-ray diffraction (XRD, see below) and the sample was used as the  $\text{FeSi}_2$  reference.

### 2.2 X-Ray Diffraction

An x-ray diffraction experiment was carried out to confirm the presence of  $\text{FeSi}_2$  evidenced through the analysis of XES measurements. Monochromatic  $\text{Cu K}\alpha_1$  (8048 eV) radiation on a Rigaku five-circle diffractometer was used to obtain the  $\theta/2\theta$  diffraction patterns of the Fe/Si multilayer and  $\text{FeSi}_2$  reference in a  $\theta$ - $\theta$  geometry in an angular scan ranging from 20 to 80° in  $2\theta$ .

The XRD patterns of the  $\text{FeSi}_2$  and Fe/Si multilayer are shown in Figure 1. A narrow x-ray diffraction peak at 45.66° is found for the  $\text{FeSi}_2$  reference. The other two peaks belong to the Si substrate [20,21]. The position of diffraction peak for  $\text{FeSi}_2$  is comparable to the diffraction peaks for  $\text{FeSi}_2$  thin film on Si substrate at 45.81° [20]. It must be noted that the comparison is only done with the diffraction peaks for  $\text{FeSi}_2$  thin films rather than the bulk values, since the lattice parameters in thin films could be different from the bulk ones. Also, considering the annealing conditions and the stoichiometry of our  $[\text{Fe}(3.6\text{nm})/\text{Si}(4.5\text{nm})]_{\times 13}$  sample we can rule out the presence of Fe and other iron silicides and can infer that the diffraction peak at 45.66° is due to  $\text{FeSi}_2$  compound only. However, for the Fe/Si multilayer,  $[\text{Fe}(2.5\text{nm})/\text{Si}(2.5\text{nm})]_{\times 40}$ , a broad peak (having a width of 1.93°) is observed at 44.66°. Given this width and closeness to both diffraction peaks of Fe and  $\text{FeSi}_2$ , from this pattern, we cannot obtain relevant information regarding the presence or not of silicide within the multilayer.



*Figure 1: X-ray diffraction patterns of the  $\text{FeSi}_2$  reference (black line) and Fe/Si multilayer (red line).*

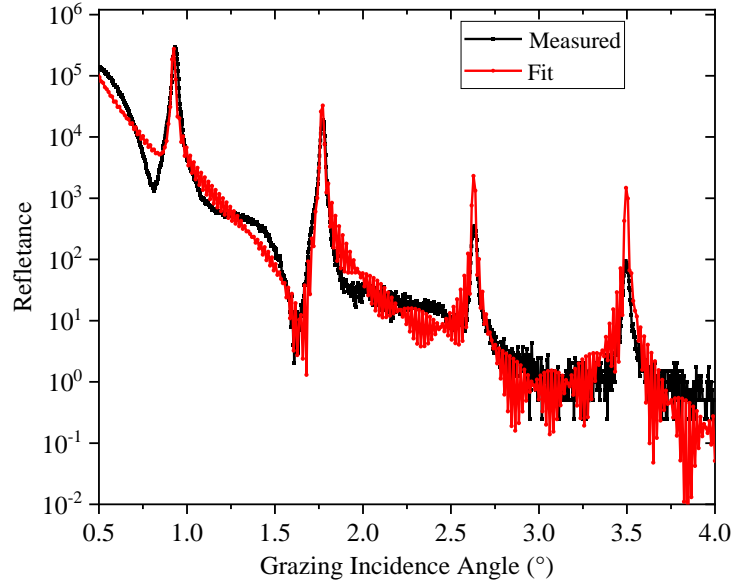
### 2.3 X-Ray Reflectivity

XRR measurements were carried out by using a Rigaku five-circle diffractometer equipped with a monochromatized Cu  $K\alpha_1$  radiation source (8048 eV). The reflectivity curve was obtained by varying the grazing incidence angle and simultaneously detecting the reflected beam in  $\theta$ - $2\theta$  geometry.

XRR is used to characterize the layers of a multilayer stack, i.e. the thickness, roughness, density and composition of the different layers and interlayers of the multilayer, provided a model of the stack giving the different layers, is defined. These parameters are calculated by fitting the experimentally obtained XRR curve of the Fe/Si multilayer using the IMD code [22]. At first, we consider a two-layer model, Fe/Si to describe the stack. Since the fit does not work well with this model we then take into account a  $\text{FeSi}_2$  interfacial layer at Fe-on-Si and Si-on-Fe interfaces, i.e. we consider a four-layer model of  $[\text{Fe}/\text{FeSi}_2/\text{Si}/\text{FeSi}_2]$  on the Si substrate. The structural parameters obtained from fitting the four-layer model of the Fe/Si stack, shown in Figure 2, are given in Table 1. The fitted density of the interlayers is close to the one of the disilicide,  $4.96 \text{ g/cm}^3$ . The deviation between the fitted and measured curves can be explained due to the uncertainty in the relative thicknesses of the layers owing to the composition gradient moving from Fe to Si layer and from Si to Fe layers.

*Table 1: Structural parameters of the layers used in the model to describe the Fe/Si stack to fit the reflectivity measurement. Layers are presented from the surface down to the substrate.*

<i>Layer</i>	<i>Thickness</i> ( $\pm 0.30 \text{ nm}$ )	<i>Roughness</i> ( $\pm 0.10 \text{ nm}$ )	<i>Density</i> ( $\pm 0.30 \text{ g/cm}^3$ )
Fe	1.78	0.67	7.86 (Bulk)
$\text{FeSi}_2$	1.10	0.35	5.09
Si	1.30	0.44	2.32 (Bulk)
$\text{FeSi}_2$	0.90	0.52	4.79



*Figure 2: X-ray reflectivity fit of the Fe/Si multilayer at 8048 eV.*

From Table 1, we can observe that the thickness of the FeSi<sub>2</sub> layer formed at the Fe-on-Si interface is slightly higher than the thickness of FeSi<sub>2</sub> formed at the Si-on-Fe interface. Even if this difference in thickness values is within the uncertainty, it could be attributed to the difference between the surface free energies of Fe (2.9 J/m<sup>2</sup>) and Si (1.2 J/m<sup>2</sup>) [23]. As the surface free energy of Si is lower than the one of Fe then during the deposition of Fe on Si, Si atoms try to move to the surface guided by the chemical driving force giving rise to intermixing between the layers. Whereas, during the deposition of Si on Fe, no such chemical driving force exists, therefore the intermixing at the Si-on-Fe interface would take place as a result of random thermal motions only [24].

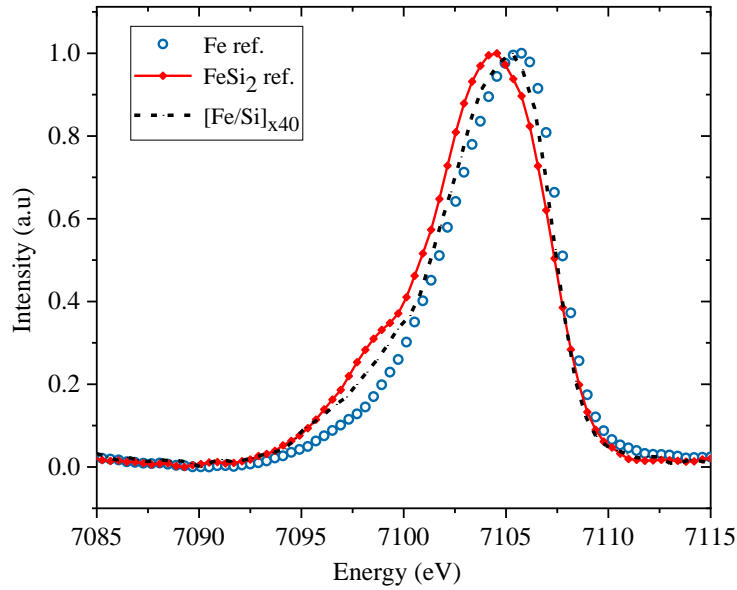
## 2.4 X-ray Emission Spectroscopy

X-Ray fluorescence analysis of the Fe K $\beta_{2,5}$  emission line in the Fe/Si multilayer, Fe and FeSi<sub>2</sub> reference samples were done at GALAXIES beamline [25] installed at synchrotron SOLEIL. The experimental set up was arranged in a Rowland geometry, where four Ge (620) analyzer crystals collect the scattered and characteristic x-rays from the sample and focus them onto a silicon drift detector. The total spectrometer resolution was estimated at 1.5 eV (full width half maximum) from the elastic line. During the experiment, it was observed that the sample surface (placed in ambient air) underwent a chemical reaction due to the formation of ozone upon irradiation by the incident beam. Hence, to prevent this reaction, the sample was placed in a helium atmosphere. This also increases the transmission of the incident and emitted radiations. The incident photon energy was 7500 eV. The data for each of these

samples was baseline subtracted and then normalized to their maximum to plot the emission curves. The Fe  $K\beta_{1,3}$  emission spectra was also measured (see supplementary information) but since the spectra of the Fe and FeSi<sub>2</sub> references were similar, not much information could be obtained about the chemical state of Fe.

### 3. Results and Discussion

To determine physical-chemical state of the Fe atoms in the multilayer, we compare the Fe  $K\beta_{2,5}$  emission spectrum of the multilayer with the ones of Fe and FeSi<sub>2</sub> references, Figure 3. The emission band results from the electronic transition from occupied 3d to 1s levels. It is a valence to core transition and is sensitive to the chemical state of emitting Fe atoms. The Fe  $K\beta_{2,5}$  emission spectra of the multilayer and the references have their own distinct shape along with a relative shift in the position of the maximum. We can also see from the Figure 3 that the Fe  $K\beta_{2,5}$  emission spectrum of the Fe/Si multilayer is located between those of Fe and FeSi<sub>2</sub> and then can be considered as a mix of the emission spectra of the two references, indicating the presence of an iron silicide at the interfaces in the multilayer stack.

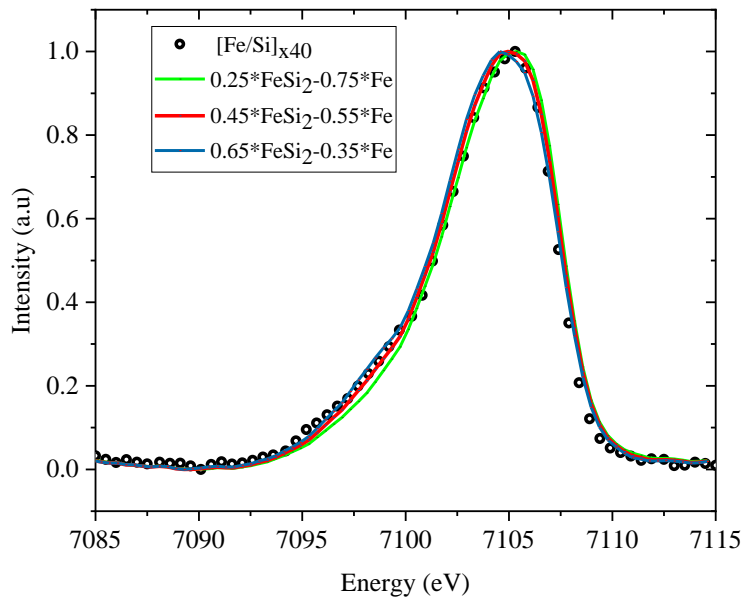


*Figure 3: Normalised Fe  $K\beta_{2,5}$  emission spectra of the Fe/Si multilayer, Fe reference and FeSi<sub>2</sub> reference.*

The spectrum of the Fe/Si multilayer is fitted with a weighted sum of Fe and FeSi<sub>2</sub> spectra. Using the weighted coefficients of Fe and FeSi<sub>2</sub>, we determine the thickness of the interfacial FeSi<sub>2</sub> layer in the multilayer stack with a model proposed by Miyata *et al.* [7]. In this model, we consider that the total Fe  $K\beta_{2,5}$  intensity emitted from the multilayer is due to the



contribution of the intensity emitted by iron atoms, present in the Fe layers and the FeSi<sub>2</sub> interfacial layers. The coefficients of the weighted fit are proportional to the emitted intensity of the respective layers, which is itself considered proportional to the thickness of each layer [7]. We also assume that the composition and thickness of the Fe-on-Si and Si-on-Fe interfaces are identical and that the densities of the reference compounds are equal to those of bulk compounds [7]. As only one XES measurement is performed it is not possible to use a more sophisticated model of the stack, where the thicknesses of the Fe-on-Si and Si-on-Fe interfaces are different and thus we assume the same thickness at both these interfaces.



*Figure-4: Fits (lines) of the Fe  $K\beta_{2,5}$  emission band of the Fe/Si multilayer (dots) by a weighted sum of the spectra of the reference compound. The best fit is obtained with 45%FeSi<sub>2</sub> + 55%Fe (thick line).*

The weighted fit of the Fe/Si multilayer spectrum is presented in figure 4. The best fit is obtained with 0.55 Fe + 0.45 FeSi<sub>2</sub>, with an uncertainty of about 0.2 on the coefficients. With 55% of FeSi<sub>2</sub> in the weighted sum, the thickness of the interfacial FeSi<sub>2</sub> is estimated to be  $1.4 \pm 0.2$  nm at all interfaces, giving a total interlayer thickness of 2.8 nm. This value is larger (40%) than the thickness deduced from the XRR analysis, 2.0 nm.

However, other silicides could also be formed at the interface, but here we stress only the application of the technique used in soft x-rays to be extended with hard x-rays. Moreover, when other silicides, FeSi and Fe<sub>3</sub>Si, are considered in the calculation of thickness whilst taking into account the bulk density for each, we find that the interfacial thickness increases to

1.5 nm and 1.6 nm, respectively, thus increasing the discrepancy between values deduced XRR and XES. Also, the growth kinetics, fabrication conditions as well as thickness deposited play a crucial role in determining the type of silicides formed at the interface. Hence, from XRR fit and XES calculations we can conclude following our model that FeSi<sub>2</sub> is formed at all the interfaces with a thickness estimated to be 1.4 nm.

#### 4. Conclusion

We studied Fe/Si multilayer by analysing the hard x-ray Fe K $\beta_{2,5}$  emission band. The characterization of the physical-chemical state of the iron atoms within the Fe/Si multilayer as obtained by XES shows the formation of iron silicide (FeSi<sub>2</sub>) of 1.4 nm thickness at the interfaces. Until now, x-ray emission has been used to study the multilayer systems in the soft x-ray range and here we presented that the same methodology could be transferred in the hard x-ray range to get information about the multilayer, qualitatively and quantitatively. This methodology could also be applied to study other multilayers provided the emission spectra for the multilayer and the associated references i.e. the possible compounds formed at the interface are of distinct shape. Furthermore, this technique gives chemically sensitive information of an element, in comparison to other methods such as XRR which is not sensitive enough to identify the small compositional gradients at the interfaces.

More precise information on the buried layers and interfaces of the multilayer stack could be facilitated by using the X-ray standing wave technique [26] combined with XES at high spectral resolution. The combination of these techniques will be sensitive to the depth distribution and chemical state of the emitting element thereby providing depth-dependent information of the multilayer in the sub-nanometer resolution.

#### Acknowledgments

Stefan Kubsky and François Nicolas from the Surface laboratory of SOLEIL synchrotron are thanked for annealing the samples. We acknowledge SOLEIL for provision of synchrotron radiation facilities (proposal 20191178).

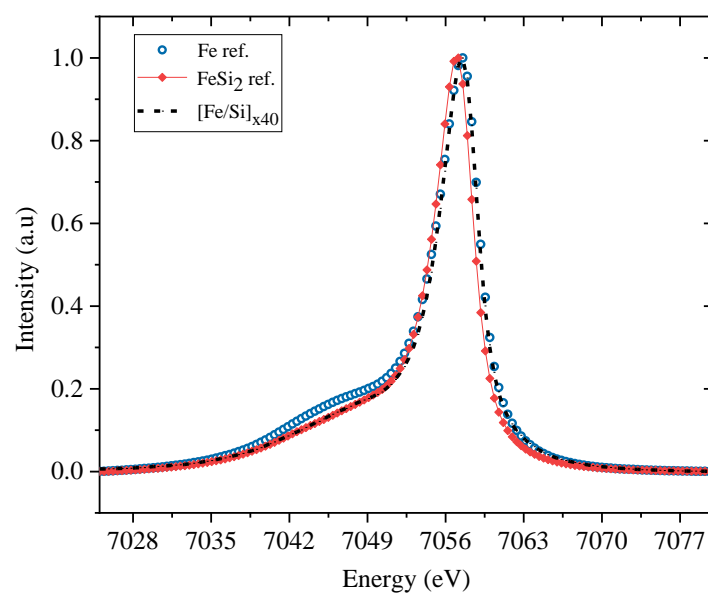
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## Supplementary Information



*Figure- Normalised Fe  $K\beta_{1,3}$  emission spectra of the Fe/Si multilayer, Fe reference and FeSi<sub>2</sub> reference.*