MÖSSBAUER EFFECT STUDY OF PROTON IRRADIATION EFFECTS IN SS-310 AND $Fe_2(SO_4)_3 \cdot xH_2O$

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ABSTRACT

The proton irradiation effects in SS-310 and $Fe_2(SO_4)_3 \cdot xH_2O$ were studied by Mössbauer spectroscopy. Irradiation aftereffects i.e. structural changes and chemical desintegration were observed and discussed.

INTRODUCTION

The hyperfine parameters to be determined from the spectra as well as the Debye-Waller factor are very sensitive concerning local conditions at the Mössbauer nucleus site and in its immediate environment. One can expect therefore that this method might be very appropriate for investigation of stable deffects appearing after irradiation.

Samples of stainless steel 310 (SS-310) and iron sulphate $Fe_2(SO_4)_3 \cdot xH_2O$ were irradiated with protons in order to investigate the influence of proton energy and beam current as well as exposure time on formation of stable defects and chemical desintegrations.

EXPERIMENTAL

Thin foils of SS-310 were irradiated in a small Cochroft-Walton generator with protons of energies 20 - 150 keV and beam currents in the range of 20 - 60 μA for 0.25^h, 1^h and 2^h. The irradiation conditions for the $Fe_2(SO_4)_3 \cdot xH_2O$ were 40 - 50 keV concerning proton energy, 40 - 50 μA beam current and 0.25^h time of bombardment. For the irradiation with 5 keV protons a mass spectrograph source was used.

Mössbauer spectra were obtained in transmission geometry with a constant acceleration spectrometer using a source of ^{57}Co in Pd. In all cases the effective absorber-thickness was 20 mg/cm² of natural iron. The velocity scale was calibrated with metallic iron. In the calculation of the Mössbauer parameters Lorentzian lines were used to fit the experimen-

tal spectra.

RESULTS AND DISCUSSION

1. Stainless steel

Some typical spectra of ^{57}Fe in SS-310 obtained before and after irradiation are shown in Fig. 1. Corresponding parameters resulting from the analysis of the spectra are given in Table I.

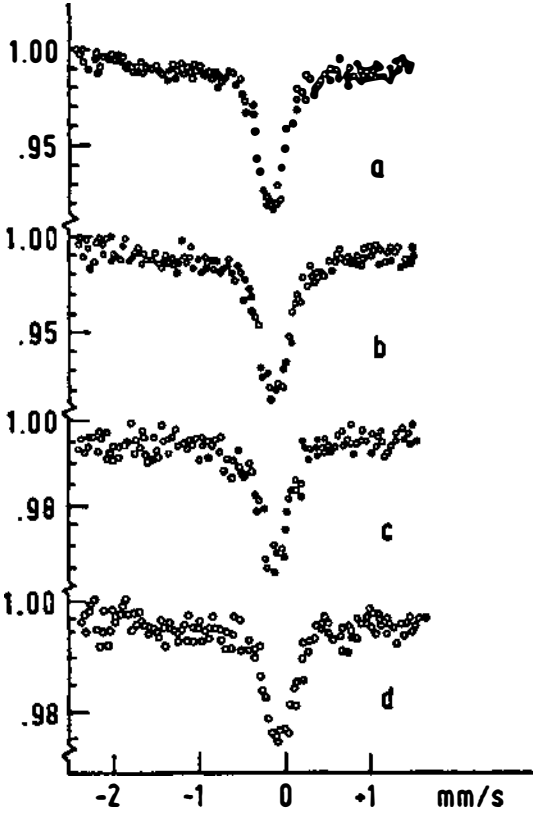

Fig. 1

Mössbauer spectra of ^{57}Fe in SS-310 at room temperature before (a) and after irradiation with protons of energy E=150 keV beam current I_p=60 μA and times of irradiation t=o.25 h(b), t=1 h(c) t=2 h(d).

Table I

Parameters of the Mössbauer spectra of ^{57}Fe in SS-310

I(μA) / E(keV)	t(h)	A	δ	Γ
nonirrad.	-	0.225	0.096	0.384
10/5	0.5	0.640	0.094	0.377
10/5	20	0.631	0.092	0.362
20/20-30	0.5	0.210		
20/50	0.5	0.198	0.096	0.368
30/100	1	0.182	0.096	0.431
50/100	0.5	0.197	0.098	0.440
60/150	0.25	0.225	0.090	0.419
60/150	1	0.115	0.108	0.424
60/150	2	0.103	0.084	0.391

A - area under the resonance line

δ - isomer shift relative to metallic iron in mm/s±0.02

Γ - width of the resonance line at half height in mm/s±0.03

These results indicate that the widths of the resonance lines in the energy region 5 - 50 keV (beam current 10 - 20 μA) decrease with the time of irradiation. At 150 keV and 60 μA the trend is reversed. The irradiation at 150 keV produced spectra with deformed base lines which could not be fitted satisfactorily by assuming Lorentzian shape.

It is remarkable that the areas under resonance lines always decrease in the energy region 50 - 150 keV. Such decrease of Mössbauer absorption can be explained by the formation of lattice deffects[1].

Nonirradiated samples exhibit a broadened singlet which is changed after irradiation in the region 20 - 30 keV at 20 μA to a doublet with parameters δ = 0.091 mm/s and ΔE_Q = = 0.181 mm/s. The width for the lower energy line is about 0.30mm/s and 0.49 mm/s for the higher one. Such change in the spectrum shape we tried to explain with the formation of lattice deffects. Using the results of the computer simulation of the effects of charge impurities upon the shape of the resonance discussed by Pustowska et all[2], we estimate that about 5 - 10 % of defects

are present.

2. $Fe_2(SO_4)_3 \cdot xH_2O$

Before the irradiation the Mössbauer spectrum of ^{57}Fe in $Fe_2(SO_4)_3 \cdot xH_2O$ is a singlet (Fig. 2) with the isomer

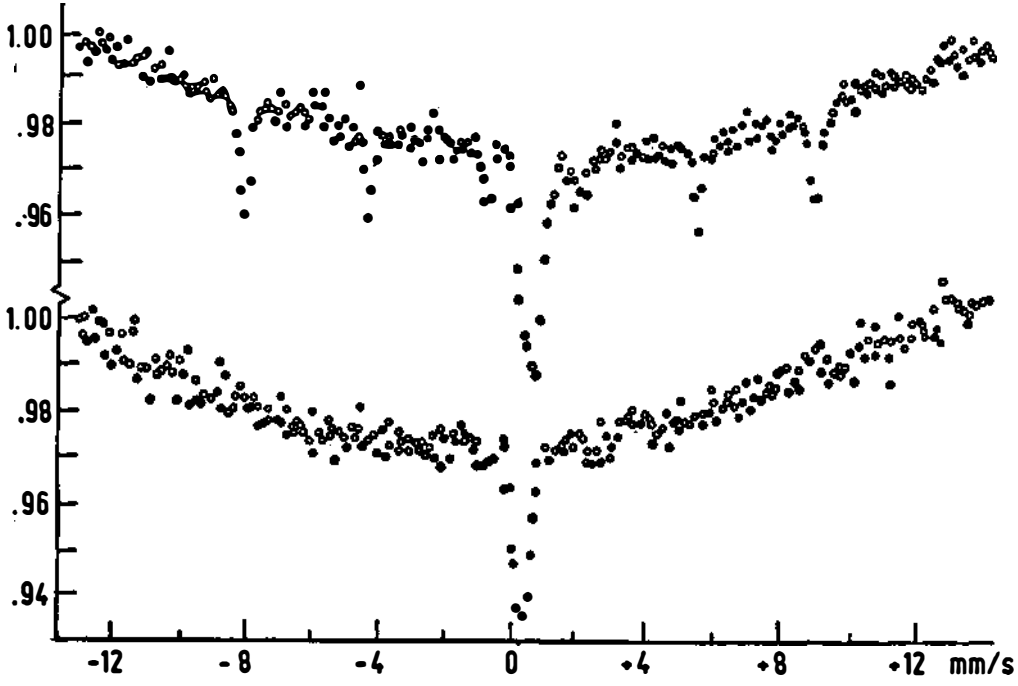

Fig. 2

Spectra of ^{57}Fe in $Fe_2(SO_4)_3 \cdot xH_2O$ before (below) and after irradiation (at the top) with protons of energy E=50 keV, beam current I_p=50 μA and the time of irradiation t=0.25 h.

shift parameter δ of 0.44±0.04 mm/s relative to metallic iron and width of the resonance line of 0.56±0.02 mm/s. After irradiation with protons in the energy region of 5 to about 40 keV and beam current of 10 - 40 μA a spectrum with the same shape but of a larger width is obtained (Table II).

The spectrum taken after irradiation for 0.25 h at 50 keV energy and current of 50 μA can be divided into a magnetically split part of lower intensity and into a singlet part of higher intensity with parameters given in Table II:

Table II

Parameters of the Mössbauer spectra of ^{57}Fe in $Fe_2(SO_4)_3 \cdot xH_2O$

Sample	$\frac{I\ (\mu A)}{E(keV)}$	t(h)	Γ	δ	ε	H	Remark
$Fe_2(SO_4)_3 \cdot xH_2O$	nonirrad.	-	0.56	0.44	-	-	singlet
"	10/5	0.50	0.62	0.41	-	-	singlet
"	50/50	0.25	0.65	0.46	-	-	singlet
				0.39	0.08	514	sextet

Γ - widths of the Mössbauer line at half height in mm/s (±0.01)

δ - isomer shift in mm/s (±0.02) relative to metallic iron

$\varepsilon = \frac{e^2qQ}{4}\left(\frac{3\cos^2\theta - 1}{2}\right)$ - quadrupole splitting parameter in mm/s

H - effective magnetic field in kgauss (±5)

The parameters for the magnetic split part of the spectrum correspond to that of iron trioxide Fe_2O_3 [3]. From the areas under the resonance lines it follows that about 45% of the ferri-sulfate transform into iron trioxide, most probably through the reaction:

$$Fe_2(SO_4)_3 \cdot xH_2O \rightarrow Fe_2O_3 + SO_2 + S + H_2O$$

Such decomposition might be due to the high temperature of about 1000 K distributed as a spike of 200 Å diameter in the case of 45 keV protons [4]. Such decomposition was observed also in the temperature controlled conditions [5].

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