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Magnetism and crystal-field properties of DyRu₂Si₂ from ¹⁶¹Dy Mössbauer spectroscopy

K Tomala^{†‡}, J P Sanchez[†] and R Kmieć[§]

[†] Centre de Recherches Nucléaires (IN₂P₃), 67037 Strasbourg Cédex, France

[§] Institute of Nuclear Physics, Cracow, Poland

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Abstract. ¹⁶¹Dy Mössbauer measurements on DyRu₂Si₂ are reported. The occurrence of a single magnetic site at low temperatures suggests that the modulated spin structure deduced from neutron data should be squared. The saturation hyperfine parameters reveal a fairly pure $|\pm 15/2\rangle$ ground state, i.e., a Dy moment of $10 \mu_B$. The unusual temperature dependence of the hyperfine field and quadrupole interaction found in the paramagnetic region is explained in the framework of a relaxation model implying excited crystal-field states. The second- (B_2^0) and fourth-order (B_4^0) crystal-field parameters are estimated to amount to -4.94 K and 0.0050 K, respectively.

1. Introduction

The intermetallic compounds RT₂Si₂ (R is a rare earth or actinide element and T a transition metal) have attracted considerable interest owing to their wide range of peculiar physical properties (Rogl 1984, Parthé and Chabot 1984). Most of them crystallise in the body-centred tetragonal ThCr₂Si₂-type structure (space group I4/mmm). The compounds usually order magnetically at low temperatures exhibiting different types of magnetic structure with magnetic moments localised predominantly at the R-site (Szytuła and Leciejewicz 1989).

The present report concerns a detailed ¹⁶¹Dy Mössbauer investigation of DyRu₂Si₂ between 4.2 and 300 K. The main purpose of this work was to shed more light on the electronic structure of the Dy³⁺ ions in this material. At low temperatures, both the magnetic hyperfine field (H_{hf}) and the electric field gradient (EFG) induced by the 4f shell electrons at the ¹⁶¹Dy nuclei are fashioned by the crystalline electric field (CEF) ground-state wavefunction. Moreover, temperature dependence of these parameters can provide information on the position and nature of the excited CEF levels.

DyRu₂Si₂ was already extensively studied by bulk magnetisation measurements and neutron diffraction (Hiebl *et al* 1983, Ślaski *et al* 1984) and some preliminary Mössbauer results were reported (Felner and Nowik 1984, Bogé *et al* 1989, Sanchez *et al* 1989). It was shown that DyRu₂Si₂ orders antiferromagnetically at 25 K with moments only localised at the Dy³⁺ ions. The magnetic structure is of sine-modulated type with a propagation vector $\mathbf{k} = (0, 0.222, 0)$ and Dy magnetic moments parallel to the tetragonal c axis.

[‡] On leave from Institute of Physics, Jagellonian University, Cracow, Poland.

The paper has been organised in the following way. In § 2 we give a brief account of the experimental details. Then we report our experimental results with emphasis on the rather unusual behaviour of the resonance spectra above the Néel temperature. In § 3, we discuss our results, propose a revised magnetic structure and present our conclusions concerning the structure of the CEF levels and the dynamics of the Dy³⁺ moments in the paramagnetic state.

2. Experimental procedure and results

2.1. Experimental procedure

Polycrystalline sample of DyRu₂Si₂ was prepared by arc melting, under argon atmosphere, of stoichiometric amounts of the constituents and subsequent vacuum annealing at 900 °C for one week. The sample analysed by x-ray diffraction using Cu K α radiation did not reveal any impurity phase. The lattice parameters measured are in good agreement with those reported previously (Hiebl *et al* 1983, Ślaski *et al* 1984).

¹⁶¹Dy Mössbauer-spectroscopy measurements were performed using a sinusoidal drive motion of a neutron activated ¹⁶⁰Gd_{0.5}¹⁶²Dy_{0.5}F₃ source kept at room temperature. The absorber was maintained at different temperatures between 4.2 and 300 K. Special care was taken to optimise the absorber thickness (≈ 13 mg of natural Dy cm⁻²) owing to the large photoabsorption cross section for the 25.6 keV gamma rays by the Ru atoms. Even then, a one week measurement for each spectrum was necessary to obtain good quality data. The gamma rays were detected using either a proportional counter (Kr-CO₂, 2 atm) or a Si(Li) high-resolution detector. The velocity calibration of the spectrometer was performed using metallic Dy ($H_{\text{hf}} = 5689(3)$ kOe, $e^2qQ = 124.9(2)$ mm s⁻¹ from NMR data, Berthier *et al* 1975). The effective-field magnetic spectra were directly least-squares computer-fitted to the hyperfine parameters by constraining the relative absorption energies and intensities of the Lorentzian lines to the theoretical values. Some spectra were analysed using spin-relaxation models (described in detail in § 3.4).

2.2. Experimental data

Figure 1 shows spectra taken at temperatures between 4.2 and 150 K. In the magnetically ordered state (i.e. at $T < 25$ K), the data are well represented by a unique set of hyperfine parameters with collinear magnetic field and EFG (table 1). The isomer shift (1.09(5) mm s⁻¹ at 4.2 K) is significantly smaller than the one observed in metallic Dy (2.88(6) mm s⁻¹). Any hyperfine parameter distribution is ruled out from the observed very narrow resonance lines ($W \approx 4.5$ mm s⁻¹).

Above T_N , the temperature dependence of the spectra clearly evidences two types of behaviour. First one should notice that neither the overall hyperfine splitting nor the spectral shape change abruptly when crossing the ordering temperature (figure 1). From T_N up to about 100 K, one observes well developed magnetic hyperfine structure with some line broadenings occurring above 77 K. The data can be adjusted either by a 'static' hyperfine Hamiltonian or by a two-level relaxation model (slow-relaxation limit) for the ground-state Kramers doublet when $77 \leq T \leq 100$ K (Wickman 1966, Wickman and Wertheim 1968). The unusual feature is that both the magnetic hyperfine field and the quadrupolar interaction decrease continuously with increasing temperature (table 1). Above 100 K, the shape of the resonance spectra is strongly modified and practically collapses to a broad single line at 150 K (figure 1). The narrow single-resonance line

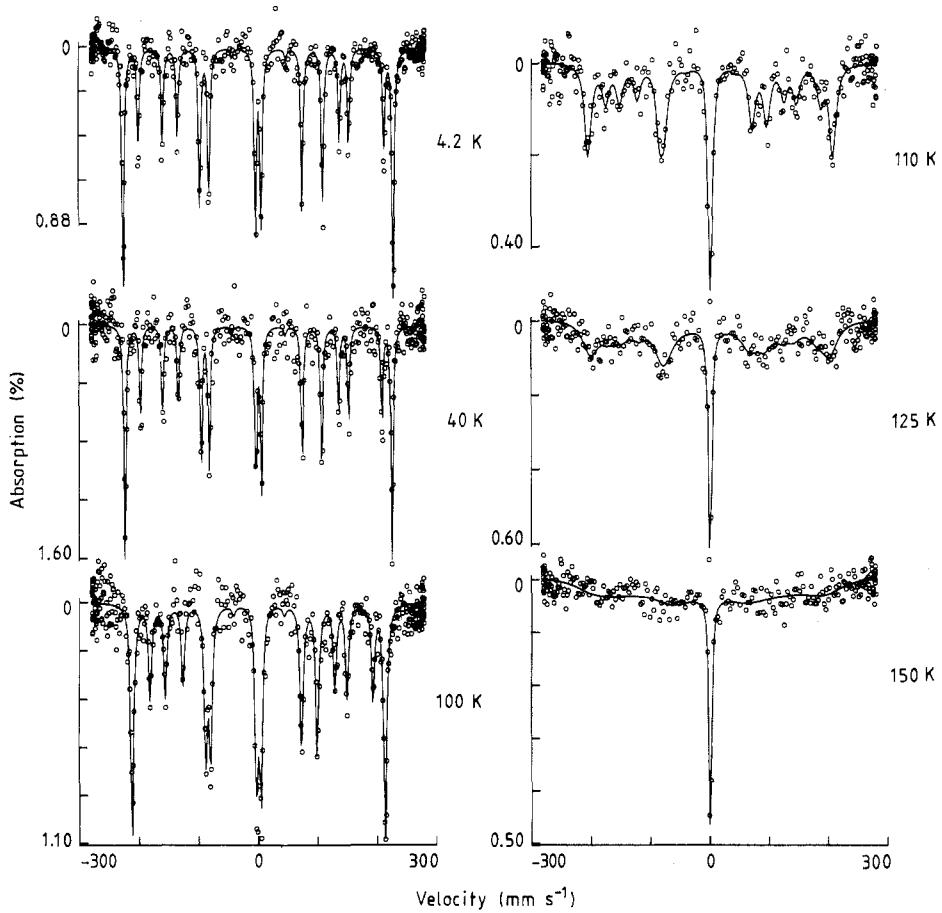


Figure 1. ^{161}Dy Mössbauer spectra of DyRu_2Si_2 at different temperatures. The spectra taken at 4.2 and 40 K were fitted using a static Hamiltonian while the data obtained at 100 K and at higher temperatures were least-squares analysed using a two-level relaxation model (see text).

Table 1. Hyperfine interaction parameters in DyRu_2Si_2 (see text).

T (K)	H_{hf} (kOe)	e^2qQ (mm s^{-1}) ^a	$H_{\text{hf}}^{\text{eff}}$ (kOe)	$e^2q^{\text{eff}}Q$ (mm s^{-1}) ^a
4.2	5720(20)	82.8(0.8)	5915(25)	133.7(5.1)
20	5744(20)	83.2(0.8)	5939(25)	134.1(5.1)
40	5668(20)	77.5(0.8)	5863(25)	128.4(5.1)
77	^b 5496(30)	65.2(1.3)	5691(35)	116.1(5.6)
100	^b 5400(30)	57.9(1.3)	5595(35)	108.8(5.6)
105.5	^b 5290(50)	52.0(4.0)	5485(55)	102.9(8.3)
110	^b 5227(50)	53.3(5.0)	5422(55)	104.2(9.3)
125	^b 5134(100)	—	5329(105)	—

^a For $E_{\gamma} = 25.6$ keV in ^{161}Dy : $1 \text{ mm s}^{-1} = 8.5576 \times 10^{-8} \text{ eV} = 20.69 \text{ MHz}$.

^b From two-level relaxation model least-squares fit.

($W = 5 \text{ mm s}^{-1}$) observed at 300 K indicates near-cancellation of the 4f and lattice contributions to the EFG. The temperature-dependent spectral shape can be well represented by a phenomenological two-level model with hyperfine parameters changing faster with temperature than in the former region and with rising relaxation rates to the fast-relaxation limit.

3. Discussion

3.1. Hyperfine field and quadrupolar interaction

The magnetic hyperfine field acting on rare-earth nuclei is commonly described as a sum of several contributions (McCausland and Mackenzie 1980)

$$H_{\text{hf}} = H_{4\text{f}} + H_{\text{cp}} + H_{\text{op}} + H_{\text{n}}. \quad (1)$$

$H_{4\text{f}}$ represents the field produced by the 4f localised electrons (orbital plus spin-dipolar contributions); H_{cp} is the core polarisation field, $H_{\text{cp}} \approx 100 (g_J - 1) J$ kOe; the last two terms stand for the conduction-electron contribution due to the own polarisation (H_{op}) and to the neighbouring magnetic atoms (H_{n}). For an S-state ion, the 4f contribution vanishes thus the $|H_{\text{hf}}|$ of 273(1) kOe measured in GdRu₂Si₂ (Czjzek *et al* 1989) provided a direct evaluation of the $H_{\text{hf}}^{4\text{f}}$ contribution in DyRu₂Si₂ when scaling the Gd data with $S = (g_J - 1)J$ factor, 5/7 (Bleaney 1972). Notice that we assumed (table 1) that H_{hf} in the Gd alloy is negative as expected when the transition metal (Ru) does not carry a magnetic moment. Besides the inaccuracy caused by this kind of scaling (owing to orbital polarisation effects, Berthier *et al* 1978), the values of $H_{\text{hf}}^{4\text{f}}$ in the paramagnetic region contain an additional error connected with the averaging to zero of the transferred field contribution (H_{n}). However, it is usually safe to assume that H_{n} is negligible when the transition metal (Ru) is non-magnetic. The saturation (4.2 K) value of $H_{\text{hf}}^{4\text{f}}$ given in table 1 compares well with the free ion $H_{\text{hf}}^{4\text{f}}$ field of 5930(30) kOe estimated from the DyAl₂ (Berthier *et al* 1975) and GdAl₂ (Cashion *et al* 1973) data using (1). Thus, the Dy³⁺ moment in DyRu₂Si₂ amounts to 10 μ_{B} .

The effective quadrupole-coupling constant e^2qQ consists of an electronic (4f) and a lattice (lat) contribution

$$e^2q_zQ = e^2q_z^{4\text{f}}Q + \frac{1}{2}e^2q_z^{\text{lat}}Q (3 \cos^2\theta - 1) \quad (2)$$

where $\theta = 0^\circ$, since the magnetic moment direction (taken as the Z axis) is parallel to the lattice EFG principal axis (z) which is along the tetragonal *c* axis.

For S-state ions (Gd³⁺) the 4f EFG vanishes to zero. This allows the lattice contribution to be calculated in the isostructural DyRu₂Si₂ from the quadrupole interaction data of GdRu₂Si₂ ($e^2qQ = -8.34(1) \text{ mm s}^{-1}$, Czjzek *et al* 1989). With the ground-state quadrupole moments $Q(^{155}\text{Gd}) = 1.30(2)b$ (Tanaka *et al* 1982) and $Q(^{161}\text{Dy}) = 2.35(16)b$ (Stevens 1981) the lattice contribution to the quadrupolar interaction at the ¹⁶¹Dy nuclei was estimated to amount to $-50.9(4.3) \text{ mm s}^{-1}$. Combining the experimental ¹⁶¹Dy e^2qQ results with the so-calculated lattice term, one obtains the electronic 4f contribution given in table 1. It should be mentioned that this procedure does not take into account the possible influence of slightly different lattice parameters.

The $e^2q^{4\text{f}}Q$ values reported in table 1 have to be compared to the free-ion value of 135(1) mm s^{-1} estimated from the quadrupolar-interaction data in cubic DyAl₂ (Berthier *et al* 1975).

3.2. Magnetic structure

As shown in § 2.2, Mössbauer spectra recorded in the magnetically ordered state can be well analysed with a single set of hyperfine parameters, thus indicating that all Dy³⁺ ions carry the same magnetic moment. Our data are in clear disagreement with the magnetic structure deduced from neutron-diffraction measurements (Ślaski *et al* 1984). Indeed, for a sine modulated structure with a propagation vector $\mathbf{k} = (0, 0.222, 0)$ one should observe in the Mössbauer spectra five different components with a large scattering of hyperfine parameters (e.g. $H_{\text{hf}} = H_{\text{hf}}^0 \cos 2\pi ky$ where H_{hf}^0 is the maximum field and y the real space distance). If the magnetic structure should be modulated we suggest a square-wave modulation which will bring in closer agreement the neutron and Mössbauer data. More accurate neutron diffraction measurements, aimed to search higher harmonics ($3k$) proving squared modulation, are highly desirable. It should be pointed out that no detectable variation of the Mössbauer spectra has been observed between 4.2 and 20 K, thus indicating that there is no evidence for a change of magnetic structure in this temperature range, i.e., sine to square modulation as observed e.g. in NdRu₂Si₂ (Chevalier *et al* 1985).

3.3. Crystal-field parameters in DyRu₂Si₂

The monotonous decrease with temperature of the magnetic hyperfine-field and quadrupole-coupling constant was attributed to thermal occupation of the excited CEF levels of the Dy³⁺ ion. The low-temperature values of H_{hf}^{4f} and $e^2q^{4f}Q$ in the paramagnetic state (table 1), found to be very close to the free ion estimates, indicate that the ground-state CEF Kramers doublet should be of almost pure $|\pm 15/2\rangle$ character.

To perform a more quantitative analysis of the data we used a truncated CEF Hamiltonian

$$\mathcal{H}_{\text{CEF}} = B_2^0 O_2^0 + B_4^0 O_4^0 + B_4^4 O_4^4. \quad (3)$$

The B_2^0 parameter was constrained to the value of -4.94 K obtained from the lattice contribution to the EFG (see § 3.1) using the relation

$$B_2^0 = \frac{-\alpha_J e q^{\text{lat}} \langle r^2 \rangle (1 - \sigma_2)}{4(1 - \gamma_\infty)} \quad (4)$$

with a selection of atomic parameter values: $(1 - \gamma_\infty) = 60$; $(1 - \sigma_2) = 0.4$; $\alpha_J = -0.0063$; and $\langle r^2 \rangle = 0.2188$ (\AA^2) (Freeman and Desclaux 1979).

The temperature dependences of H_{hf}^{4f} and $e^2q^{4f}Q$ in the range between 20 and 100 K were simultaneously least-squares fitted by numerical diagonalisation of the CEF Hamiltonian using one set of B_4^0 and B_4^4 parameters. B_4^0 was found to be equal to $0.0050(6)$ K and the fitted curve was shown to be insensitive to the value of the B_4^4 coefficient. An independent check of the influence of B_4^4 was performed by changing B_4^4 step-by-step between -0.01 K and 0.01 K. It was shown that neither the eigenvalues nor the eigenfunctions were significantly modified. It is therefore concluded that, owing to the anomalously large B_2^0 term, the CEF splitting is dominated by the axial part of the CEF Hamiltonian giving almost pure $|\pm m\rangle$ Kramers doublets with the ground state being $|\pm 15/2\rangle$. It is also demonstrated that the Dy moment direction is strongly correlated to the negative sign of B_2^0 , which forces the easy axis of magnetisation to be along the tetragonal c axis. The results of the least-squares fit to the $H_{\text{hf}}^{4f}(T)$ and $e^2q^{4f}Q(T)$ data are shown in figure 2, together with the proposed level scheme of the low-energy Kramers

doublets. Independent fits of the $H_{\text{hf}}^{\text{4f}}(T)$ and $e^2q^{\text{4f}}Q(T)$ data slightly improve the agreement between calculated and experimental points giving $B_4^0 = 0.0050(7)$ K and $B_4^0 = 0.0055(3)$ K, respectively.

3.4. Dynamics of the Dy^{3+} magnetic moment

In the previous section we showed that the Dy^{3+} ions experience magnetic hyperfine interaction up to about 150 K, i.e., well above the Néel temperature. It was also pointed out that the spectral shape is not strongly modified by dynamical processes between T_{N} and 100 K but the hyperfine-interaction parameters (H_{hf} and e^2qQ), given straightforwardly by the positions of the rather sharp resonance lines, decrease monotonously with increasing temperature.

The behaviour outlined above was already observed in DyCo_2Si_2 (Asch *et al* 1983). The authors explained the unusual decrease of H_{hf} and e^2qQ with temperature as being due to a slight variation of the CEF parameters that influence the character of the ground-state Kramers doublet. A possible cause of this effect could be a temperature-dependent c/a ratio. Thus, their low-temperature spectra were described by a two-level model (ground-state Kramers doublet) in the limit of slow relaxation.

It seems that such an explanation does not apply to DyRu_2Si_2 . Indeed it was shown that B_2^0 is anomalously large, thus it is hard to influence the nature of the ground-state wave function by changing higher-order CEF parameters. Therefore we suggest another relaxation mechanism able to explain the temperature dependence of H_{hf} and e^2qQ .

In § 3.3 we gave arguments that the ground and low-lying excited CEF states should be of almost pure $|\pm m\rangle$ character. Neither spin–lattice nor spin–spin interactions can induce electronic relaxation within the ground-state $|\pm 15/2\rangle$ Kramers doublet (Nowik 1967). With some caution the same argument may be applied to the next $|\pm 13/2\rangle$ and $|\pm 11/2\rangle$ doublets. Nevertheless transitions induced by spin–spin relaxation between states with $\Delta m = \pm 1$ are allowed and expected in concentrated magnetic systems. Thus our physical system can be divided into two thermodynamic subsystems characterised by $|+m\rangle$ and $|-m\rangle$ states, respectively. Notice that the two subsystems with the same inner-spin dynamics give identical Mössbauer spectra. Transitions between the two subsystems are forbidden (at least below 100 K) whereas fast fluctuations may occur within each subsystem. Thus, the well resolved Mössbauer spectra observed in the paramagnetic state (from T_{N} up to 100 K) can be understood as resulting from fast electronic fluctuations between either $|+m\rangle$ or $|-m\rangle$ CEF levels. Fast relaxation allows the average values of hyperfine interaction parameters which were discussed in § 3.1 to be introduced. This relaxation mechanism is reminiscent of the well known ‘ferromagnetic relaxation’, which is applied here to a paramagnet with a special character of the CEF splitting.

The analysis of the resonance spectra taken between T_{N} and 100 K was performed in the framework of the above relaxation model applying the Wickman’s formalism (Wickman 1966) with relaxation matrix truncated to transitions between the two or three lowest CEF levels. Higher CEF levels, lying above 350 K, are expected to have little influence on the spin dynamics below 100 K. Each $|+m\rangle$ or $|-m\rangle$ level contributes its characteristic hyperfine-interaction constants (estimated from free-ion values) weighed by the Boltzmann factor, and only transitions which fulfil the $\Delta m = \pm 1$ selection rule are allowed. The linewidth of the resonance was constrained to 4.5 mm s^{-1} , the value obtained from the spectrum taken at 4.2 K. Then the spectral shape will only be influenced by the relaxation rate Ω and the energies E_1 and E_2 of the excited levels (see figure

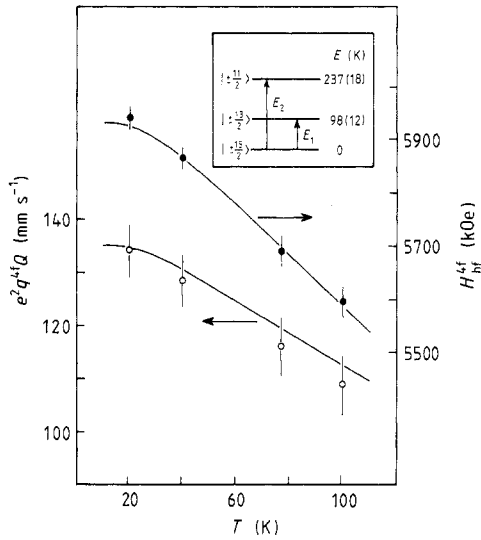


Figure 2. Temperature dependence of the 4f contribution to the hyperfine field and quadrupole interaction. The curve to the data points corresponds to a simultaneous fit with CEF parameters given in the text. The inset represents the CEF-level diagrams obtained from the data analysis.

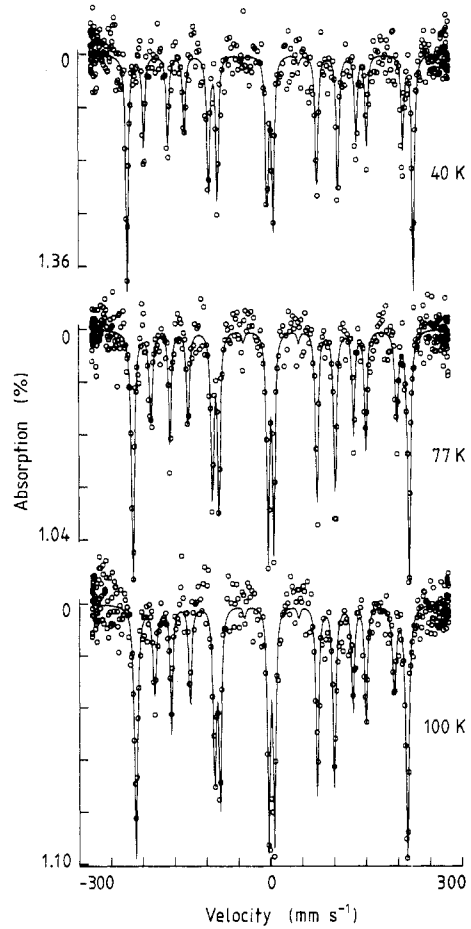


Figure 3. ¹⁶¹Dy Mössbauer spectra of DyRu₂Si₂ taken in the paramagnetic region and fitted using the relaxation model described in the text and with parameters given in table 2.

2). It was found that the spectral shape (being in the fast relaxation limit) is rather insensitive to the actual value of the relaxation rate. The energy of the first excited state E_1 can be estimated straightforwardly from the analysis of the 40 K spectrum when more highly excited levels are almost unpopulated. Spectra shown in figure 3 were analysed with $\Omega = 2000 \text{ mm s}^{-1}$ ($\approx 40 \text{ GHz}$) and excitation energies given in table 2. Since spin-spin interactions are mainly responsible for the dynamics of the Dy moments; Ω was expected to be temperature-independent in the temperature range considered.

As shown in figure 1, the spectral shape changes abruptly above 100 K; this behaviour was attributed to the occurrence of spin-lattice relaxation via Orbach and Raman processes (Orbach 1961) that are able to induce transitions between both subsystems of the CEF levels. The misfit observed in the central part of the 100 K spectrum (figure 3) already indicated that 100 K is at the borderline where spin-lattice relaxation starts to be effective. The resonance spectra can be reproduced by a phenomenological two-level

Table 2. Excitation energies and average values of hyperfine parameters obtained from least-squares fit of the Mössbauer data using the relaxation model described in § 3.4. Average values of H_{hf} and e^2qQ were calculated from fitted excitation energies E_1 and E_2 and adjusted hyperfine interaction constants for ground and excited CEF levels (see text).

T (K)	E_1 (K)	E_2 (K)	$\langle H_{\text{hf}} \rangle$ (kOe)	$\langle e^2qQ \rangle$ (mm s ⁻¹)
40	99(15)	—	5675(15)	79.9(1.5)
77	99 ^a	187(20)	5482(35)	66.7(2.8)
100	99 ^a	181(20)	5387(35)	60.4(2.8)

^a Fixed value in the fitting procedure.

relaxation model, but one should rather understand them as resulting from slower relaxation between both subsystems and fast relaxation inside each of them.

4. Summary and conclusions

The present work reports the results of a ¹⁶¹Dy Mössbauer study of DyRu₂Si₂ in the temperature range from 4.2 to 300 K. The observation of a single magnetic site in the ordered phase suggests a square-wave modulated spin structure instead of a sine modulation as concluded from neutron-diffraction data. The analysis of the saturation hyperfine-field and quadrupole-interaction data points to a fairly pure $|\pm 15/2\rangle$ electronic ground state. The occurrence in the paramagnetic region (from T_N up to 100 K) of 'static' magnetic hyperfine spectra with temperature-dependent hyperfine parameters was explained in a straightforward way using a relaxation model (fast-relaxation limit) involving excited crystal-field levels and magnetic fluctuations mainly due to spin–spin interactions. This model allowed information on the nature and energies of the CEF states to be obtained. It was further shown that the sign (negative) of B_2^0 determines the easy magnetisation direction (tetragonal c axis) and thus the single-ion anisotropy. The rapid collapse of the magnetic hyperfine structure above 100 K was attributed to the increase of the relaxation rate due to spin–lattice processes.

Acknowledgments

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