Methods of Spectral Estimation in Local Nuclear Quadrupole Resonance with a Dispersion

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Received January 23, 2002

Abstract—The spectral estimation in local nuclear quadrupole resonance at a high noise level is performed for the first time using the modern techniques of linear prediction (LPSVD) and matrix pencil (ITMPM). The fast Fourier transform with signal accumulation does not ensure the required sensitivity in the case of weak signals when the object and the receiver of the spectrometer are spaced widely apart or when there is an effect of adverse factors (screening, interference, random disturbance, etc.), which is typical of remote monitoring in actual practice. It is demonstrated that the use of the proposed techniques considerably increases the efficiency of spectral estimation in this field of solid-state spectroscopy and, in particular, avoids the phase errors arising in usual experiments at a signal-to-noise ratio of less than 0.5.

In recent years, local nuclear quadrupole resonance (NQR) has found wide use in detecting and identifying different technological mixtures, including explosives and narcotics [1, 2]. As a rule, the detection of signals at a distance of longer than 5 cm from the studied sample is performed at a signal-to-noise ratio (SNR) of less than unity and, hence, necessitates additional processing of the signals.

In the general case, the pulsed nuclear quadrupole resonance is characterized by a decaying cosine signal, which can be represented in the form

\[
S(t) = A e^{-\omega T_2} \cos[2\pi f_c t + \varphi(t)].
\]  

(1)

Here, \(T_2\) is the spin–spin relaxation time [for the hexogen (RDX) explosive at 300 K, \(T_2 = 1.5\) ms], \(f_c\) is the frequency of nuclear quadrupole resonance, and \(\varphi(t)\) is the phase of the signal distorted through narrow-band additive noise, which is described by the function

\[
n(t) = x(t)\cos2\pi f_c t - y(t)\sin2\pi f_c t.
\]

(2)

The noise function can be represented by the relationship

\[
n(t) = n_x(t)\cos[2\pi f_c t + \varphi(t)] - n_y(t)\sin[2\pi f_c t + \varphi(t)],
\]

(3)

where

\[
n_x(t) = x(t)\cos\varphi(t) + y(t)\sin\varphi(t),
\]

(4)

\[
n_y(t) = -x(t)\sin\varphi(t) + y(t)\cos\varphi(t).
\]

After multiplying the signal with noise \(s(t) + u(t)\) in a synchronous detector by the reference voltage \(c(t) = \cos[2\pi f_c t + \varphi(t)]\), we obtain

\[
e(t) = A e^{-\omega T_2} \cos\varphi + n_x(t)\sin\varphi + n_y(t)\cos\varphi,
\]

(5)

where \(\varphi = \varphi - \varphi\) is the phase error.

The phase error is determined by the signal-to-noise ratio. The performance of the synchronous or quadrature detector is adversely affected if the signal-to-noise ratio is less than a certain value (SNR < 0.5), and the upset of synchronization with subsequent accumulation gives rise to impulse noise. If the phase of the reference voltage is stable and is specified by the voltage across the synthesizer, the phase of the signal with noise at a smaller signal-to-noise ratio involves error, which leads to a decrease in the accumulation efficiency. In the case when the signal-to-noise ratio is equal to 0.5, the phase error reaches 60° and causes a signal power loss of 2.5 dB. For the purpose of optimizing this process, it is necessary to choose appropriately both the frequency of detuning \(f_c\) between the synthesizer and NQR frequencies at a specified distance from the surface coil and the phase of the reference voltage across the synchronous detector.

In order to solve the problem of spectral estimation in local nuclear quadrupole resonance, we used the modern methods of linear prediction (LPSVD) and matrix pencil (ITMPM) [3] with the Mathlab V5 program (see table); as far as we know, these methods were not applied earlier for this purpose. Prior to inputting into a Pentium 900 PC, the NQR signals were digitized using a sound card (2046 points).

As can be seen from the table, the LPSVD and ITMPM methods offer approximately the same results...
for spectral estimation in the local nuclear quadrupole resonance, thus providing a considerable increase in the information content and the detection probability (success rate) to 97–99% within the 25-cm range, which is substantially higher than that in other methods (mass spectrometry and X-ray diffraction analysis).

In our NQR spectrometer, the phase errors with an increase in the distance are eliminated using a phase shifter [4–6]. This makes it possible to observe the NQR signals from a 450-g RDX sample at a distance as long as 35 cm. It should be noted that no significant distortions in the spectral line of nuclear quadrupole resonance in RDX are revealed in our experiments. This is inconsistent with the results obtained by Anferov et al. [7]: instead of one line at a frequency of 5192 kHz, they observed two or even four lines when employing the fast Fourier transform in the absence of phase coherence of the pulses. Since the phase errors arise from the decrease in the signal-to-noise ratio with variations in the distances in local nuclear quadrupole resonance, the use of phase pulse sequences and composite pulses to distort the spectral line through noise becomes inexpedient.

An NQR signal can be represented as a set of absorption signals \( V(f_d) \) and dispersion \( w(f_d) \) which are characterized by different dependences on the frequency of detuning \( f_d \). The quadrature detector can extract a pure absorption signal if the phase \( \varphi = 0 \) for one channel and \( \varphi = \frac{\pi}{2} \) for the other channel. In the general case, we have \( V^2(f_d)\cos^2\Delta\varphi + w^2(f_d)\sin^2\Delta\varphi \neq 1 \): i.e., the phase error is not compensated and a distorted signal is observed in both channels. The operation with detuning in the SQRC program [5] becomes difficult because the detuning cannot be optimum. Figure 1 displays a pure \(^{14}\text{N}\) absorption signal extracted with the use of a synchronous detector at a frequency of 5192 kHz in hexogen (a 450-g sample is located at a distance of 1 cm from the surface of the coil). Figure 2 depicts an NQR signal in RDX at a signal-to-noise ratio of 0.5. The obtained parameters are presented in the table. It follows from the table that the parametric methods have certain advantages when operating under noise. In local nuclear quadrupole resonance, the signal-to-noise ratio decreases in inverse proportion to the distance from the sample; i.e., at a distance of 25 cm, the signal-to-noise ratio decreases by a factor of 25 and amounts to 0.2. Therefore, the use of the LPSVD and ITMPM methods at a signal-to-noise ratio of 0.5 necessitates preliminary signal accumulation (no less than ten accumulations) provided the distance exceeds 12 cm.

With the aim of decreasing the effect of acoustic resonances arising in different plastic parts, we applied the quantum beat method described by Kirchanov et al. [8] and Proakis [9]. The transitions at frequencies \( v_0 = 1782 \) kHz and \( v_r = 3410 \) kHz were excited with the use of two crossed semitoroidal coils and the radiation at the frequency \( v_r \) (5192 kHz) was received into a ring 20 cm in diameter that was located normally to the fields of the coils. This also appreciably decreased the insensitive time during transition at the \( v_r \) frequency.

For a small signal-to-noise ratio, the dependence of the phase error on the signal-to-noise ratio exhibits a nonlinear behavior and the variance of the phase error is determined by the formula \( \sigma_{\varphi}^2 = \frac{1}{\text{SNR}} \). The phase error probability with a change in the distance from the material to the surface coil [9] can be estimated from the expression

\[
P(\Delta\varphi) = \frac{\exp(\text{SNR}\cos\Delta\varphi)}{2\pi I_0(\text{SNR})} ,
\]

where \( I_0 \) is the zero-order Bessel function.

**Fig. 1.** An NQR signal in hexogen at a frequency of 5192 kHz.