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Current density imaging by pulsed conduction electron spin resonance

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Abstract

In analogy with Nuclear MRI, the ESR signal phase shift of conduction electrons moving in electrical currents along controlled magnetic field gradients can be used to generate spatial *electronic current* density maps. First two-dimensional images of the current density distribution in quasi-one-dimensional organic conductors are presented.

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1. Introduction

Electron-Spin magnetic resonance imaging (ESMRI) is conceptually almost identical with nuclear magnetic resonance imaging (NMRI) in that they both involve the detection of spins in magnetic field gradients. Due to the comparatively fast electron spin relaxation times, ESMRI is technically demanding and therefore much less developed. Nevertheless, a variety of experimental schemes have been already adapted from NMRI and the number of applications appears to be growing steadily [1–4]. Thus, focussing on *conduction* electrons in particular, some recent studies used ESMRI to investigate the statistical electron motion, i.e. electronic diffusion (see for example [5,6]). In these cases, the locally mapped spin echo time-decay under applied gradients was investigated.

In connection with one of the most important manifestations of conduction electrons—namely electrical current—further analogy to NMRI comes to mind: Using phase contrast NMRI to measure material flow is a well-known practice developed extensively for noninvasive quantitative imaging of *in vivo* blood flow, yielding many of the clinical procedures of MRI angiography [7,8]. The basic principle [9,10] involves monitoring the phase shift of nuclear spins attached to molecules that move with the liquid along a magnetic field gradient in combination with spatial resolution obtained by standard imaging techniques. In this work, we present the adaption of the phase-contrast method to conduction electrons in order to visualize *electrical current density* distribution in solids.

2. Experimental details

2.1. Imaging scheme

For an introduction to NMRI, we refer to standard textbooks [11,12]. The principles of scanning $\vec{k}$-space [13]

$$\vec{k} = \frac{1}{2\pi}\gamma \int \vec{G}(t)\,\mathrm{d}t \tag{1}$$

(with gyromagnetic ratio γ and applied magnetic field gradient $\vec{G}$), known from NMRI, can be directly adapted to electron spin resonance imaging. Scanning the k-space in a cartesian grid and deriving spin density images by performing inverse Fourier transforms is the well-known so called Fourier Imaging.

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In order to detect the conduction electron flow resulting from (or leading to) a constant dc current I, we use a modification of a basic flow-encoding building block [11]. Beside the standard spin echo excitation sequence $\pi/2$–τ–π–τ-echo and a set of phase encoding gradients with variable strength to implement the Fourier Imaging, this sequence contains a quasi-static gradient which is constant at least during the whole excitation-acquisition period, starting at $t < 0$ and ending at $t > 2\tau$. Such quasi-static gradient is depicted as G_x on the left hand side in Fig. 1. Assuming stationary spins, the quasi-static gradient effects a dephasing during the first part of the sequence which is compensated by the same gradient after applying the refocusing π-pulse, leaving the phase of the signal at the center of the spin echo, at 2τ, unchanged. But in the presence of a DC electronic current, with the spins acquiring a constant component of velocity along the gradient direction x, the position of the electron spins and therewith the spatially depending magnetic field strength as well as their Larmor frequency vary with time, resulting in a phase shift of the central echo signal given by

$$\Delta\phi(2\tau) = -\gamma G v \tau^2. \quad (2)$$

Using Eq. (2) for $\Delta\phi$ leads to a systematic error, as it is accurate for the scheme depicted in Fig. 1 only in the limit of vanishing width for the $\pi/2$ and π RF pulses. An accurate expression can be easily derived for the more common 'modified bipolar' scheme with finite RF pulse-widths, provided G_x is kept off for the duration of the RF pulses [11]. However, the benefit of thus eliminating the small systematic error in $\Delta\phi$ is more than compensated by the technical simplification and the increased sensitivity offered by the sequence of Fig. 1.

It should be noted that the quasi-static gradient G_x is not used, in this case, as a classic read gradient to obtain spatial resolution, but rather as a phase encoding gradient. Thus, only the $t = 2\tau$-data point is analyzed. A crucial point in the practice of detecting *electrical* currents by phase contrast experiments arises because of the additional magnetic field gradients generated by the current itself. To eliminate these additional gradients, a difference experiment was developed. The experiment is repeated twice, once with G_x and another time with inverted gradient polarity $-G_x$. In both experiments, a phase shift, $\Delta\phi^+$ or $\Delta\phi^-$, respectively, is detected (Fig. 1). Because the current induced magnetic field gradients are unchanged by the inverted G_x gradient, their influence on the phase shift is annulled by subtracting the signals of both experiments. Thus corrected, the phase shift can be written as

$$\Delta\Phi = \Delta\phi^+(2\tau) - \Delta\phi^-(2\tau) = -2\gamma G v \tau^2 \quad (3)$$

choosing $\Delta\Phi \equiv 0$ for $I = 0$.

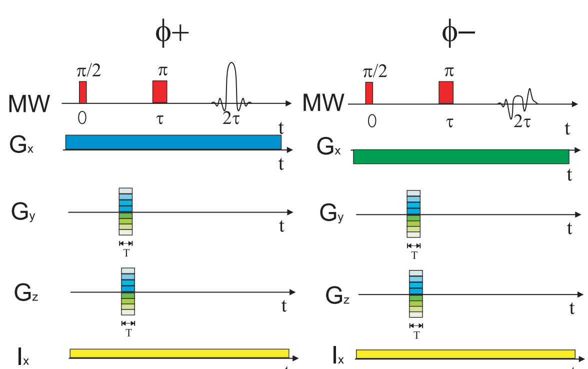

Fig. 1. Schematic of the pulse sequence containing pulsed Fourier imaging gradients $G_{y,z}$ as well as a "static" gradient G_x resulting in a current induced phase shift. $G_{y,z}$ were stepped in both polarities to scan data points in two-dimensions of k-space. Two experiments, ϕ^+ and ϕ^-, with inverted current encoding gradient are performed in order to eliminate additional, unwanted current induced gradients. The spin echo phase shift was determined at $t = 2\tau$.

2.2. Experimental realization

Detecting the small phase shifts expected and found in our case dictates a S/N ratio which is attainable only by performing the pulsed ESR in the microwave frequency range rather than in the lower radio-frequency range available in standard NMR imagers. The pulsed ESR imaging results were obtained with a commercial Bruker Elexsys E580 spectrometer at approximately 9.3 GHz, using an ER 4118X-MD4 dielectric ring resonator installed inside an ER4118CF continuous flow cryostat. For the experiments described below, liquid nitrogen was used as cooling agent. The $\pi/2$–τ–π–τ-echo pulse sequence was applied with a typical length of 16 ns for the $\pi/2$-pulse and using a 16-step phase cycle [11]. Hall probe regulation of the magnetic field strength of the electromagnet, cooled via heat exchanger, provided sufficient long-time stability.

The standard Bruker set-up has been equipped additionally with a home-built three-dimensional gradient system to generate the pulse sequence described above, as well as a special sample holder used to provide the electrical current through contacted samples inside the cavity. We use a coordinate system defined by the horizontal polarizing external field (z), the vertical cavity access axis (y) and the third perpendicular direction (x). A quadrupolar gradient coil, mounted around the adapted outer shell of the cryostat, enables the application of quasi-static gradients in the x-direction reaching $G_x \leqslant 0.2$ T/m for periods of at least 2 ms. To obtain the required short gradient pulses at the sample position, both G_y and G_z had to be realized with coils situated inside the dielectric ring resonator. The pulsed G_z-gradient is generated by Anti-Helmholtz gradient coils developed and delivered by Bruker, and inserted inside the ER 4118X-MD4 dielectric ring resonator. The gradient intensity amounts to $G_z/I_{G_z} = 0.66$ T/m/A. The pulsed gradient G_y is implemented with a pair of "8" shaped 0.1 mm thick copper loops of about 7 mm total extension (Fig. 2), fixed with two component epoxy resin between two 50 μm thick plastic films and introduced into the inner bore of the G_z-coils after being bent into cylindrical form. The coils are situated more than 5 mm apart from the metallic walls of the cavity [11,14].