

Comparison of 2D-ESR Filtered Back Projection and Fourier Imaging techniques in two different frequency ranges

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Introduction

There are two popular techniques for Electron Spin Resonance (ESR) imaging. While the *Filtered Back Projection* (FBP) works exclusively with static but variable gradients, *Fourier Imaging* (FI) can be performed with static phase encoding gradients known as Single Point Imaging (SPI) or, like in NMRI, with a combination of static and pulsed gradients which leads to a much quicker measurement procedure compared to SPI. The main problem concerning this technique are the very short T_2 relaxation times of biologically viable ESR spin probes (some μ s) compared to NMR which demands extremely short gradient pulses for phase encoding and which restricted FI with pulsed gradients to some rare exceptions up to now [1]. On the other hand FBP is an easy to implement and to control technique. But large static gradients lead to short T_2 and reduce the image quality as well. An additional degrading factor when working in the radio frequency range are the relatively long dead times of the spectrometers. In order to compare and evaluate these two fundamental methods we developed four pulsed 2D ESR imaging systems which allow high resolution FBP and FI images of the same sample in two frequency ranges (radio frequency (370 MHz) and X-band (9.5 GHz)). As a model system the well described one-dimensional conductor (Fluoranthene)₂PF₆ [2] was chosen, since the relaxation time is of the order $T_2 \approx 10 \mu$ s ($\sigma_{\parallel} \approx 100 \text{ S cm}^{-1} > 10^3 \sigma_{\perp}$).

Methods

For the radio frequency range all measurements were performed with a Tecmag APOLLO spectrometer operating at 370 MHz and a minimum pulse duration of 300 ns. Because of the small sample dimensions (<1.5 mm) home-made RF saddle coils of 1-2 mm diameter and only 4 windings were used [3]. The desired rotating gradient for FBP was generated by two perpendicular home-developed gradient assemblies (an anti-Helmholtz coil pair and a second order quadrupole coil) with a maximum strength of 80 mT/m at constant operation, allowing polar k-space sampling. For FI a home-built pulsed current driver and a small anti-Helmholtz coil pair produced gradient pulses with a duration of only 2.8 μ s (slew rate > 130,000 mT/m/ms). The X-band measurements were performed with a Bruker ELEXSYS ESP E580 ESR spectrometer operating with a cavity at 9.6 GHz which allows minimum microwave pulsewidths of 4 ns. Appropriate soft iron wedges installed between the pole caps of the electromagnet provided static gradients of up to 2200 mT/m in a fixed direction. For FBP the sample had to be rotated around the axis perpendicular to that gradient field while for FI a Bruker pulse coil and a current driver similar to the one built for radiofrequency FI was used. Running standard spin-echo sequences ($\pi/2-\tau-\pi-\tau$), echo times (TE) < 1 μ s could be used for X-band measurements in contrast to RF measurements where TE was in the order of 6 μ s (and therefore in the range of T_2) due to the much longer dead time of the spectrometer. For image reconstruction two C based software packages were developed, involving interpolation from a spherical to a Cartesian grid in the case of FBP. Before FT is performed multiple corrections and filters (baseline, bandwidth, phase, etc.) are available to influence the reconstruction process.

Results

In Fig. 1 typical images of various (FA)₂PF₆-samples can be seen. Fig. 1a) shows a 2D image reconstructed with FBP in the RF range as well as the optical photography, verifying the metrics of the sample (acquisition time = 35 min). A typical image for pulsed FI can be seen in Fig. 1 b) showing two (FA)₂PF₆ samples (total measurement time = 15 min). Comparing the two imaging methods, in both frequency ranges FBP leads to images of superior quality compared to pulsed FI. Especially the longer echo times and the influence of eddy currents may be reasons for this observation. Compared to the field strength in the RF range, the higher B_0 -field in the X-band leads to an improved S/N and allows the application of stronger gradients which leads to a much better image quality and physical spatial resolution (2D) which was around 10 μ m in the X-band and 25 μ m in the RF range (sample thickness = 400 μ m). A clear disadvantage of higher frequencies appears when thicker samples are examined and the B_1 -field loses intensity during the penetration into the sample. The so called "skin-effect" could lead to misinterpretations of spin density maps which is demonstrated in Fig. 1c) and 1d). Fig. 1c) displays two samples, imaged in the X-band with FBP ($G = 490 \text{ mT/m}$). The larger of the two crystals shows a "hole" artefact in the area of the thickest part of the sample. This can't be recognized in Fig. 1d) where the same crystal is imaged in the RF range (FBP, $G = 40 \text{ mT/m}$). Using pulsed ESR techniques, it is also possible to analyze the diffusion and relaxation mechanism by acquiring a series of images over an extended pulse separation τ range. Fitting that series of images, pixel by pixel, to the appropriate relaxation- or diffusion function respectively, T_2 and diffusion maps could be obtained [4].

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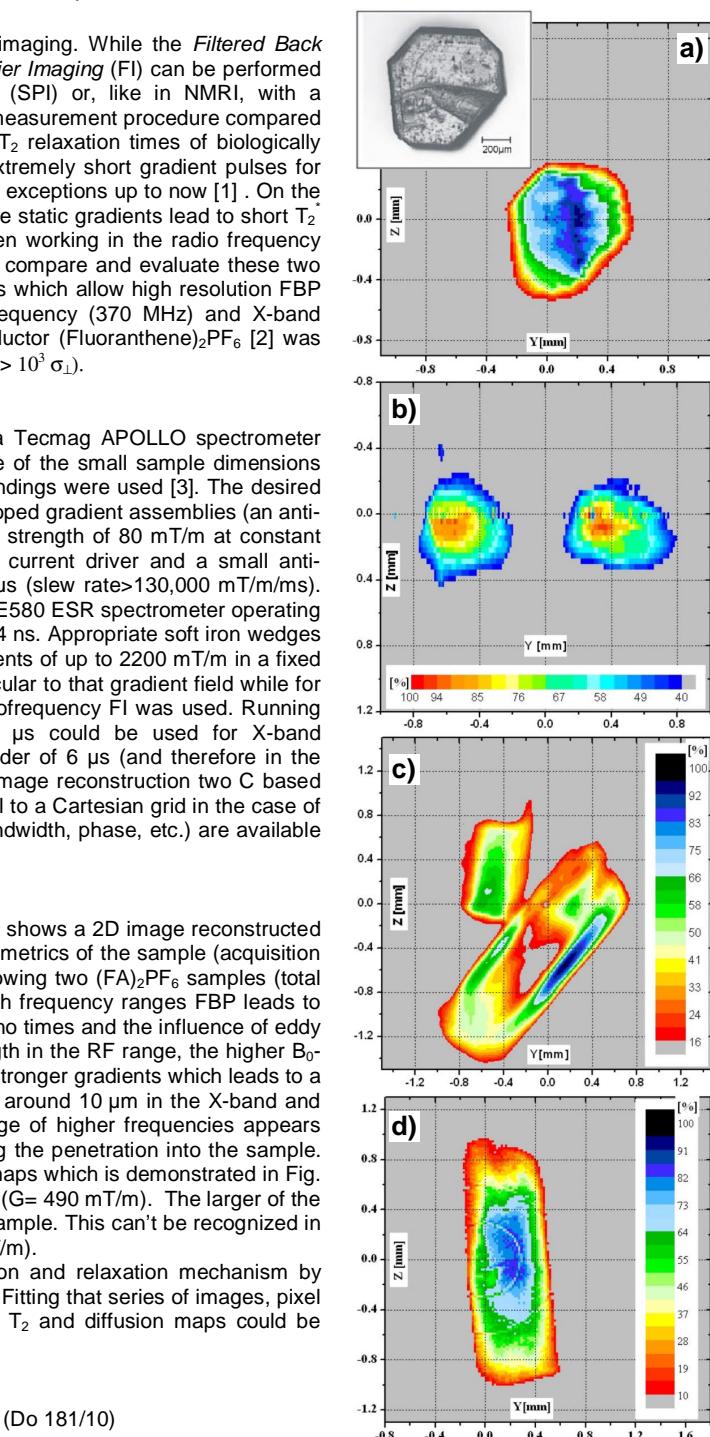


Fig.1 Various (FA)₂PF₆ 2D imaging tests ($T = 250 \text{ K}$): a) Radiofrequency FBP image ($G = 40 \text{ mT/m}$, 25000 scans per 1.4°) with optical photography. b) Two FA₂PF₆ crystals mapped with pulsed FI at 370 MHz (64 phase encoding steps, $G_{\text{read}} = 40 \text{ mT/m}$). c) X-band FBP image of two FA₂PF₆ samples ($G = 490 \text{ mT/m}$) showing the strong influence of the "skin-effect" ("hole" artifact). d) Same thick crystal shown in c) but here with FBP ($G = 40 \text{ mT/m}$) in the RF range.