Experimental report

28/10/2015

Proposal:	**5-42-366**	**Council:**	4/2014
Title:	Spatially resolved studies of metastable and ground state vortex lattice domain dynamics in MgB2		
Research area:	Physics		

This proposal is a new proposal

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Samples: MgB2

Instrument	Requested days	Allocated days	From	To
D33	5	3	03/12/2014	06/12/2014

Abstract:

The MgB2 vortex lattice (VL) exhibits significant metastability between highly ordered but differently oriented configurations. The metastability has been clearly demonstrated not to be due to vortex pinning, and we have instead proposed that it may be attributed to VL domain jamming.
This jamming hypothesis is supported by our recent measurements using an AC magnetic field to drive the VL to the ground state. This show a cross-over from a slow to a fast power-law behavior, which is speculated to be due to ground state domain nucleation and growth respectively.
Here we propose to perform spatially resolved SANS studies to investigate the dynamics of the transition to the VL ground state. Preliminary measurements have demonstrated the feasibility of such measurements, which we will develop further using the D33 multi-beam configuration.

Experiment Summary for Proposal 5-42-366: Spatially resolved studies of metastable and ground state vortex lattice domain dynamics in MgB_2

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INTRODUCTION

Previous SANS studies of the vortex lattice (VL) phases in MgB_2 have revealed an unprecedented degree of metastability that is conclusively not due to vortex pinning [1]. This metastable state (MS) can be created by field cooling or warming across any of the phase boundaries, but most often it is prepared by cooling across the F-L phase, see Fig. 1. The VL can be driven from the MS to the ground states (GS) by a small AC magnetic field. This transition takes the form of a stretched exponential as a function of the number of applied AC cycles: $(A\ e^{-(\frac{x}{\tau})^{\beta}} + C)$. The stretching exponential, β, is either approximately 1 (a regular exponential) or monotonically increases with AC amplitude depending on whether the AC field is oriented parallel or perpendicular to the applied DC field.

The goal of this experiment was to determine if there were any correlations between the VL state and real space. Spatially-resolved SANS measurements were performed by using micrometer sized apertures and scanning across the sample.

Additional measurements were performed at the ILL following a different, unsuccessful proposal (Proposal 5-42-388 on $Sn_{0.9}In_{0.1}Te$). During this beamtime, the amount of VL disorder throughout the transition from MS to GS was examined. The width of a rocking curve (RC) is indicative of the correlation length (ζ_L) along the vortices. To determine if the VL transition results in any fracturing of vortices, RCs were performed as the VL was driven to the GS.

EXPERIMENT DETAILS

The experiment was performed on the D33 beam line at the ILL using the standard SANS configuration. The specific instrument settings used for the experiment are listed in Table I.

The primary MgB_2 single crystal was mounted on a 6 mm diameter sapphire disk adhered to a thin aluminum plate. This aluminum plate had a small hole for the sample and was painted with gadolinium oxide to reduce background scattering. The sample was centered within C. Dewhurst's copper coil, which is capable of generating a perpendicular AC magnetic field on the order of a few mT. The entire coil setup was then attached to the end of a sample stick for a cryomagnet, see Fig. 2.

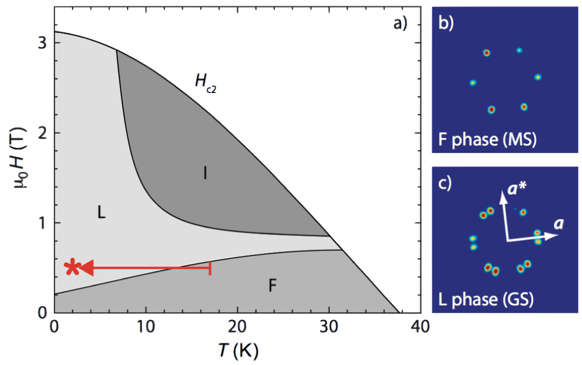

FIG. 1: (a) The GS VL phase diagram consists of three hexagonal phases: F, L, and I. The F and I phase diffraction patterns have six peaks (b) and the L has 12 (c) [2]. Field cooling at 0.5 T across the F-L phase boundary to 2 K, as shown by the red arrow, creates a MS VL.

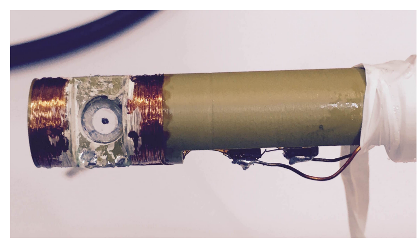

FIG. 2: The MgB_2 crystal mounted on a sapphire disk, in between an AC magnetic field coil.

Parameter	Value
Wavelength (λ)	7 $\AA$
$\Delta\lambda/\lambda$	10%
Temperature	2 K
DC Field	0.5 T
Collimation	12.8 m
Detector Distance	13 m
Source Aperture (Raster Scan)	20 mm
Source Aperture (Rocking Curves)	10 mm
Sample Aperture (Raster Scan)	200, 100 μm
Sample Aperture (Rocking Curves)	3 mm

TABLE I: D33 Instrument Settings

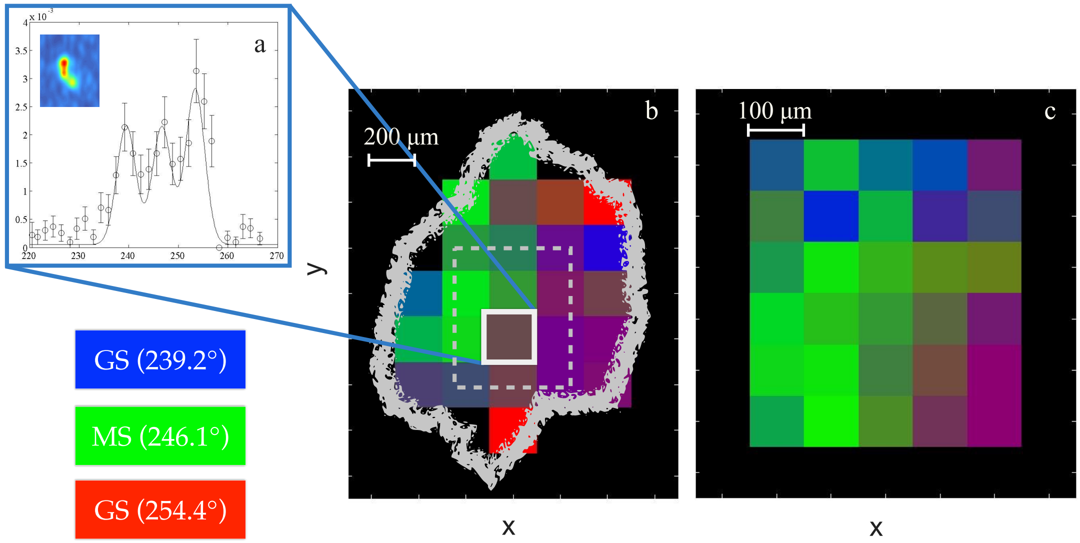

FIG. 3: The f_{MS} for each pixel of the raster scans was determined from Eq. 1 and the azimuthal distribution of intensity (a), where the inset depicts the original diffraction pattern. The resulting distribution of states can be visualized with an RGB color scale (b), where each color represents a different state – GS_1, MS, or GS_2. The central region of the sample, as indicated by the dashed rectangle, was examined further with a 100 μm aperture raster scan (c). Overall, the 200 μm (b) and 100 μm (c) scans exhibit the same general features.

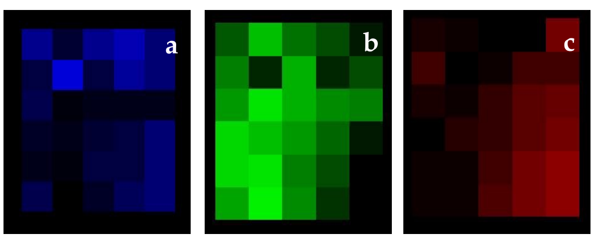

FIG. 4: The 100 μm raster scan (Fig. 3c) decomposed into individual colormaps for the GS_1 (a), MS (b), and GS_2 (c) domains.

MEASUREMENTS AND RESULTS

For all measurements, the metastable VL state was prepared by cooling in a field of 0.5 T from 20 K to 2 K across the F-L phase boundary. To ensure that the F phase was well ordered before crossing the phase boundary, a 0.5 T damped DC field oscillation was performed. The fractional metastable volume (f_{MS}) was calculated in the usual manner:

$$f_{MS} = \frac{I_{MS}}{I_{GS_1} + I_{MS} + I_{GS_2}} \tag{1}$$

where I_{GS_1}, I_{MS}, and I_{GS_2} are the integrated intensities for each state. These are determined by fitting the azimuthal distribution of the diffraction pattern with three gaussian peaks.

Spatial Correlation

A MS VL was prepared, and then driven to a bulk state of approximately equal intensities ($f_{GS_1} = f_{MS} = f_{GS_2} = 33\%$) with an AC field of 7 mT and 250 Hz. After which, a 200 μm sample aperture was used to perform a raster scan with a step size of 200 μm. For each raster scan, the sample is scanned in real space, taking a diffraction pattern at each step or "pixel". The central region of the sample was then examined further by repeating the raster scan with a 100 μm aperture and matching step size. Apertures of this size image an area of the sample containing on the order of 10^6 vortices.

The f_{MS} in each pixel can be determined as outlined above, and then visualized with an RGB colormap where each color represents a different state. These real space distributions for both aperture sizes can be seen in Fig. 3. The states are also plotted independently (one colormap for each state) in Fig. 4. Overall, the two colormaps exhibited the same general features. Few pixels were composed of entirely one state, but there are distinct regions of the sample that are predominantly MS or GS.

Longitudinal VL Correlation

After prepping the MS, an AC field of 7 mT and 250 Hz was applied for some number of cycles. A diffraction pattern was measured to determine the change in f_{MS}. This process was repeated for an exponentially increasing number of cycles, until the VL would not transition

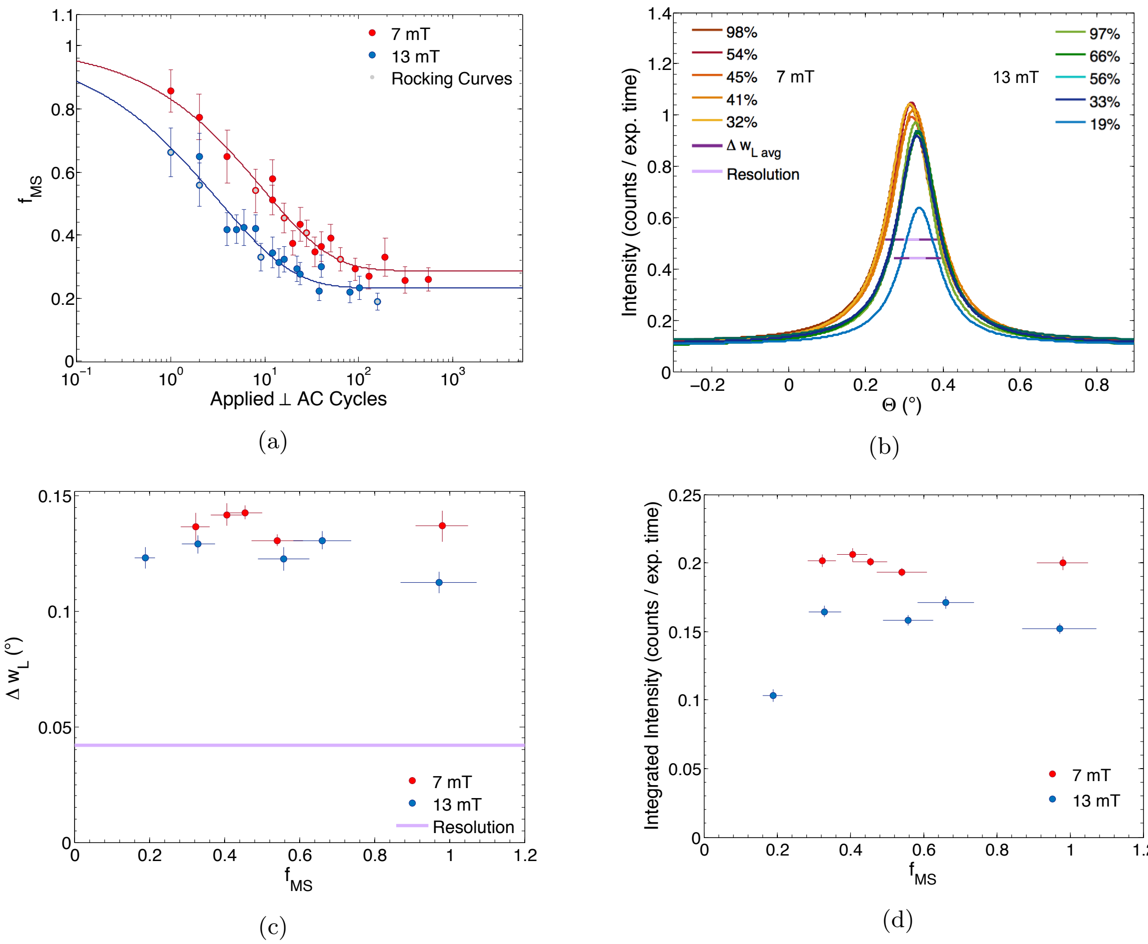

FIG. 5: (a) The system was driven from the MS to the GS with successive application of AC field cycles, with gray symbols indicating points along the transition where a rocking curve was performed. These rocking curves (b) are almost identical, with the exception of the 13 mT, 19 % curve. There was no broadening (c) throughout the transition, nor does there seem to be a correlation between the integrated intensity and MS volume fraction (d).

further. RCs were performed at several locations along the stretched exponential decay, as indicated in Fig. 5a. The entire procedure was then repeated for an AC field amplitude of 13 mT.

The RCs can be seen in Fig. 5b. From the fits, the full width half max (Δw_L) and integrated intensities were extracted and plotted as a function of f_{MS}, Figs. 5c and 5d. With the exception of the $f_{MS} = 19\%$ RC, all of the curves are essentially identical with similar integrated intensities. Furthermore, there was no correlation between the f_{MS} and either Δw_L or the integrated intensity. This lack of azimuthal broadening indicates that, within experimental resolution, there was no significant disordering of the VL.

CONCLUSIONS

From the raster scans, almost every pixel of the colormap contained more than one state, limiting the domain size to be less than 100 μm. From the rocking curves, no measurable broadening was observed and the correlation length along the vortices (ζ_L) was found to be comparable to the crystal thickness.

$$
\begin{aligned}
\Delta\omega_L &= (0.13 \pm 0.01)^\circ = (2.3 \pm 2)\ mrad \\
\zeta_L &= (\pi\ \Delta\omega_L)^{-1} = 1.4 \times 10^2\ a_0 \quad (14\ \mu m)
\end{aligned}
\tag{2}
$$

[1] Rastovski, C. et al. Persistence of Metastable Vortex Lattice Domains in MgB_2 in the Presence of Vortex Motion. Phys. Rev. Lett. 111, 107002 (2013).

[2] Das, P. et al. Observation of Well-Ordered Metastable Vortex Lattice Phases in Superconducting MgB_2 Using Small-Angle Neutron Scattering. Phys. Rev. Lett. 108, 167001 (2012).