Magnons in the Multiferroic Phase of Cupric Oxide

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Polarized neutron scattering is used to characterize low-energy magnetic excitations in the multiferroic and antiferromagnetic phases of cupric oxide (CuO). The experiments are performed with the novel type of polarization analysis available at the thermal three-axes spectrometer PUMA@FRM II allowing the simultaneous detection of spinflip and nonspinflip scattering. The energy gaps of several magnon modes are determined, and evidence for the existence of electromagnons near 3 and 13 meV is found.

1. Introduction

Even if cupric oxide (CuO) has a rather simple chemical composition, it nevertheless attracted a lot of attention during the 1980s and 1990s as the last century model substance for high-temperature superconductors. The interest faded away after a while, but since the discovery of spontaneous electric polarization in one of its antiferromagnetic phases, CuO became again a focus of research as the model substance for high-temperature type-II multiferroics.

CuO exhibits three successive antiferromagnetic phase transitions at \( T_{N1} = 213 \text{ K} \), \( T_{N2} = 229.2 \text{ K} \), and \( T_{N3} = 229.8 \text{ K} \). These temperatures are considerably higher than those in most other type-II multiferroics, making CuO quite unique as a promising stepping stone in the search for room-temperature multiferroics. The transition at \( T_{N1} \) is of first-order nature, while second-order transitions take place at \( T_{N2} \) and \( T_{N3} \). All three antiferromagnetic phases are denoted with increasing temperatures as AF1, AF2, and AF3. Details about the magnetic structures of AF1 and AF2 are explained in Section 2. The AF3 phase, on the contrary, was discovered only recently, and its magnetic structure is not yet determined.

Ferroelectricity was found to exist only in the AF2 phase with a spontaneous polarization of \( P = 160 \mu \text{C m}^{-2} \) along the \( b \)-axis. The magnetic structure of this phase is characterized by a proper spin cycloid. CuO is therefore another example of type-II multiferroics whose ferroelectricity is induced by a nonlinear spin arrangement due to anisotropic superexchange interactions, often referred to as Dzyaloshinskii–Moriya interaction. This is supported by the fact that it is possible to switch the chirality of antiferromagnetic domains with different spin orientations by an external electric field.

The determination of magnetic excitations by inelastic neutron scattering can be helpful to further investigate the microscopic mechanism of multiferroicity, especially because a low-energy electromagnon was discovered in the AF2 phase by THz spectroscopy. Electromagnons are magnetic excitations with partially phononic characters. Similar to the phenomenon that static magnetic order may induce spontaneous electric polarization, the oscillation of the magnetic structure can induce an oscillating electric dipole field. Electromagnons are therefore regarded as elementary excitations involving multiferroic coupling. As optical THz spectroscopy can only probe the \( \Gamma \) point of the magnon dispersion, inelastic neutron scattering provides a unique option for complementary investigations addressing the behavior within the entire Brillouin zone. Moreover, polarization analysis allows the distinction of magnetic and nuclear excitations and the determination of the respective eigenvectors.

Most inelastic magnetic measurements which have been published so far were, however, carried out either at temperatures well below \( T_{N2} \) or in the paramagnetic phase above \( T_{N2} \). In any case, very steep magnon branches were detected which could only be resolved with characteristic slopes of several 100 meV Å\(^{-1}\). Electromagnons, however, have been predicted in an energy range below 20 meV and one particular mode of this type has been identified at 2.9 meV using optical THz spectroscopy. The current study is focused on the low-energy behavior (<25 meV), the determination of energy gaps for different magnon modes, both in the AF1 and AF2 phases, and the possible identification of electromagnons by neutron scattering with improved resolution in momentum–energy space. Details of the magnon dispersion, on the contrary, are beyond the scope of this article. For our experiments, we used the thermal neutron three-axes spectrometer PUMA@FRM II with its novel type of polarization analysis equipment. This method proved to be a powerful tool not only for the characterization of the eigenvector of magnons but also for saving precious beam time because both spin states are measured simultaneously. We restricted ourselves to neutron polarization parallel or antiparallel to the magnetic \( b \)-direction, of the CuO sample, rather than attempting to provide a full 3D analysis.

Parallel to our own study, Jacobsen et al. independently performed similar experiments using conventional methods of polarized neutron scattering. They were able to determine details of the exchange interactions, and their results are consistent with...
our own findings. However, due to the improved experimental resolution used in the present study, we could identify additional features of different magnon branches in the low-energy regime.

2. Crystal and Magnetic Structure of CuO

CuO crystallizes in the monoclinic space group $C2/c^{(21)}$ with lattice parameters $a = 4.6927(4)$, $b = 3.4283(4)$, $c = 5.1288(6)$, and $\beta = 99.546(9)^{229}$. There are four chemical formula units per crystallographic unit cell. Cu$^{2+}$ has $3d^9$ electron configuration with total spin of $S = 0.5$ and is therefore prone to Jahn–Teller displacement. This is reflected by a highly distorted octahedral coordination geometry of surrounding oxygen ions where the average bond length in the basal plane is considerably smaller $(\approx 1.96 \text{ Å})$ than the distance to apical oxygen atoms $(2.78 \text{ Å})$. The coordination can, therefore, rather be described as rectangles of oxygen atoms that form infinite edge-shared chains (see Figure 1, left). There are two types of chains propagating in different directions, $[1 0 1]$ and $[1 0 −1]$, respectively. The bonding between the neighboring Cu atoms is mediated via oxygen atoms, forming infinite $…-\text{Cu--O--Cu--O}…$ zig-zag chains in $[1 0 1]$- and $[1 0 −1]$-directions. The Cu--O--Cu bond angles vary significantly for both types of chains, with $108.4^\circ$ along $[1 0 1]$ and $146.2^\circ$ along $[1 0 −1]$. Both types of bonds are represented on the right-hand side of Figure 1 by yellow and red tubes, respectively.

The magnetic structure of CuO in its low-temperature phase was discovered in a series of neutron scattering studies$^{[14,23–26]}$ and differs between the AF1 and AF2 phases. Figure 2 is a schematic representation of the copper atoms (red circles) in four crystallographic unit cells with their corresponding spins (blue arrows). In the low-temperature AF1 phase, all magnetic moments are oriented either parallel or antiparallel to the $b$-axis, forming a magnetic superlattice represented by the commensurate modulation vector $q_{\text{c}} = (0.5, 0, −0.5)$. Along the $[1 0 1]$-direction, all spins are ferromagnetically correlated, whereas along $[1 0 −1]$, the antiferromagnetic order is established. On entering the multiferroic AF2 phase, an additional magnetic component emerges along the direction $(0.506 a^* + 1.517 c^*)$, which will from now on be called “easy-axis.” The structure is incommensurately modulated according to $q_{\text{IC}} = (0.506, 0, −0.483)$ with a mismatch vector $\delta q = (0.006, 0, 0.017)$ almost parallel to the easy-axis. Along $[1 0 1]$, the spins are no longer ferromagnetic but experience a slight phase shift from one Cu atom to the next which results in a cycloid rotation in the plane spanned by the $b$-axis and the easy-axis which will further on be called “cycloidal plane.” This plane can also be described by its normal vector $S_{\text{b}} \times S_i$ which is parallel to the magnetically least favored direction, the “hard-axis.” According to Ain et al.$^{[26]}$, the envelope of the spins is represented by an almost perfect circle.

3. Results and Discussion

Different scans were performed close to the magnetic satellite positions $q_{\text{c}}$ or $q_{\text{IC}}$ in different Brillouin zones of the parent lattice in a temperature range covering the AF1 and AF2 phases. Of particular interest are the $(0 0 2)$ and the $(−2 0 2)$ zones since here, the scattering vector $Q$ is almost parallel to the easy-axis and the hard-axis, respectively, as shown in Figure 2. The comparison of spinflip (SF) and nonspinflip (NSF) intensities in both Brillouin zones allows us to distinguish between different types or eigenvectors of magnetic excitations because the scattered intensity is determined by the component of magnetic moments perpendicular to the scattering vector $Q$. Therefore, in the $(−2 0 2)$ zone, we observe intensity from magnetic moments or their fluctuations which are confined to the cycloidal plane spanned by the monoclinic $b$-axis and the easy-axis. The same selection rules apply for the $(0 0 2)$ zone. Here, inelastic scans are impossible for kinematic reasons, but elastic scans are favored by the strong increase in the magnetic form factor. Moments along $b$ do not affect the spins of the neutrons, whereas components along the easy-axis lead to SF scattering.

In the $(0 0 2)$ zone, on the contrary, SF scattering is associated with fluctuations of the magnetic moments along the hard-axis perpendicular to the cycloidal plane.

![Figure 1. Crystal structure of CuO with top view of the ab-plane (left) and approximately along the b-axis (right). Blue and green spheres are Cu$^{2+}$ ions with different relative coordinates. In the left panel, green and blue Cu ions have $z = 0$ and $z = 0.5$, respectively. In the right panel, green and blue Cu ions have $\gamma = 0.25$ and 0.75, respectively. Red spheres are oxygen atoms. Orange planes in the left panel represent oxygen coordination rectangles. Yellow and red tubes in the right panel represent Cu--O--bonds in [1 0 1]- and [1 0 −1]-direction, respectively. Crystallographic data for this figure are taken from a previous study$^{[229]}$.](image-url)
3.1. Elastic Measurements

As a reference, elastic scans across the magnetic satellites have been performed as a function of temperature. The corresponding intensities of the commensurate ($q_C$) and incommensurate ($q_{IC}$) satellite for SF and NSF scattering are shown in Figure 3.

The results are in agreement with a previous study by Forsyth et al. [23]. Below 213 K, there is only intensity of the commensurate satellite in the NSF channel because all magnetic moments are well aligned along $b$. With the onset of the AF2 phase at $T_{N2}$, the commensurate satellite disappears, and both SF and NSF intensities are observed to increase at the incommensurate satellite position. In a small temperature interval of about 2 K, both phases coexist which reflects the first-order nature of this phase transition.[3,4,14,27,28] Within the multiferroic AF2 phase, the incommensurate intensity decreases continuously on heating just like electric polarization.[1,4,12] Near $T_{N2} = 230$ K, the intensity fades away due to the second-order nature of this transition.[4,14] Some residual intensity is, however, still detectable at 233 K. In a previous study, it was reported that the satellite reflection persists even up to temperatures of 400 °C.[24] In our experiments, this observation could not be reproduced.

3.2. Magnon Spectra

Because magnon branches are extremely steep in the AF1 phase[14–17] as well as in the AF2 phase[20], it is not possible to resolve the $q$ dependence of the magnon energy close to the magnetic zone center at $q_{C/IC}$. A constant energy scan leads to a broad intensity distribution with the center at the position of the superlattice reflection and an increasing half-width at higher energies. This is not only true for the antiferromagnetic AF1 phase but also for excitations in the multiferroic AF2 phase. We therefore conducted constant $Q$ scans (see Figure 4) at the position of the respective satellites whereby the intensity at a given energy corresponds to magnon branches propagating in all directions.

Similar to the elastic measurements, scans in two different Brillouin zones help distinguish between modes of different eigenvectors. As shown in Figure 4a,b for the AF1 phase at 200 K just below the phase transition, there is only background intensity for the NSF channel in both Brillouin zones. As all spins in this phase are oriented along the $b$-axis, the eigenvectors of any magnon mode are restricted to the scattering plane, thus leading to SF scattering only. Within the (0 0 2) Brillouin zone (Figure 4a), the intensity starts to increase rapidly above 3 meV, which indicates that there is a spin gap in the magnon dispersion below this energy. This is followed by a gradual decrease in intensity after a maximum around 5 meV, which can be explained by resolution effects as well as the large but finite slope of the magnon branches.

![Figure 2](image1.png)

**Figure 2.** Magnetic structure of CuO in the AF1 (left) and AF2 phase (right). View is along the $b$-direction onto the $a$–$c$-plane. Red circles represent copper atoms and blue arrows the corresponding spins. Four crystallographic unit cells are shown for each phase. The orientation of the scattering vectors in the (0 0 2) and (−2 0 2) Brillouin zone and the respective direction of magnetic polarization of the SF channel are shown by black and blue arrows, respectively.

![Figure 3](image2.png)

**Figure 3.** Temperature dependence of the integrated intensity of magnetic superlattice reflections in Brillouin zone (0 0 0) in different phases. Lines are drawn to guide the eye. Reproduced with permission.[19] Copyright 2018, Elsevier.
The spectrum in the (2 0 –2) Brillouin zone is similar (see Figure 4b) but shows a smaller spin gap. In this case, the maximum intensity is already reached around 2 meV. This finding is consistent with the fact that the fluctuations probed in this Brillouin zone are along the easy-axis, whereas in the (0 0 2) Brillouin zone, only fluctuations along the hard-axis are visible.

Within the multiferroic AF2 phase with a cycloidal spin arrangement, the magnon spectra exhibit a different behavior: The SF spectrum in the (0 0 2) Brillouin zone (see Figure 4c) is similar to that of the AF1 phase with a maximum around 3 meV and a gradual decrease in intensity with higher energy transfer. This mode can be associated with a fluctuation of spins orthogonal to the cycloidal plane (tilt mode). The NSF channel, however, does not reveal any intensity maximum and therefore no spin gap within the experimental resolution of about 1 meV. This mode corresponds to spin displacements along the b-direction. The lack of a spin gap can also be found in both spin channels of the spectrum in the (2 0 –2) Brillouin zone (see Figure 4d). Here, the NSF intensity corresponds to the component of the spin fluctuations, whereas the SF channel is attributed to the component along the easy-axis.

SF and NSF channels exhibiting no spin gap correspond to the same mode, which is a sliding mode of the spin cycloid. At the magnetic zone center, this is equivalent to a phase shift of the whole cycloid with respect to the crystallographic lattice and therefore called phason. This is an energy-free excitation in the case of a regular circular cycloid like CuO \cite{26}, explaining the lack of a spin gap. Both intensities in Figure 4d are in fact equal due to the almost circular shape of the spin cycloid. This kind of magnetic excitation is unique for materials with cycloidal spin order and was first described by Katsura et al. \cite{29} A phason mode was identified by polarized neutron scattering in TbMnO$_3$ which has a similar magnetic structure like CuO \cite{30}.

There are two other possible magnetic excitations of a spin cycloid which both involve the rotation of the cycloidal plane along different axes perpendicular to the cycloidal rotation vector $S_s \times S_p$ \cite{29,30}. This implies in both cases a displacement of spins perpendicular to the cycloidal plane, being responsible for the SF intensity of Figure 4c. The spin gap of 3 meV corresponds to the energy of the magnon branch at the magnetic zone center which is in accordance with the energy of an electromagnon found in CuO \cite{13}. In that publication, THz spectroscopy was used which is an optical method that probes the magnetic zone center. A theoretical investigation of the electromagnon \cite{18} came to the conclusion that this mode is the rotation of the cycloidal plane along the easy-axis. The second mode, rotation along the b-axis, was also predicted to be an electromagnon at 13.5 meV.

More information about the magnetic excitations in the AF2 phase can be gained from constant energy scans across the satellite position. In Figure 5, SF and NSF intensity profiles are shown at 218 K for different energies (5, 10, and 25 meV) in the Brillouin zone (2 0 2) \cite{31}. Although no difference between both polarizations is expected for a bare phason mode as discussed previously, we observe that the SF intensity is significantly larger than the NSF intensity at higher energies.

**Figure 4.** SF (red) and NSF (black) intensity for constant Q scans at the magnetic superlattice reflection in both antiferromagnetic phases: a,b) AF1 and c,d) AF2 and two different Brillouin zones: a,c) (0 0 2) and b,d) (2 0 –2).
Whether this finding is due to an elliptical distortion of the spin cycloid or additional magnon–phonon interactions cannot be clearly decided on the basis of the present data and requires more detailed investigations of the dispersion relations over a larger $q$ range, considering the existence of the spinon continuum.

The NSF profile is characterized by a well-defined splitting of the peak at 25 meV which corresponds to the dispersion of the magnon branch. In contrast, the SF profile can still be represented by a single Gaussian with an increasing line width. This indicates that there are additional modes which contribute to the SF intensity at higher energies. Obviously, the dispersion of the corresponding magnons is steep enough that no distinct splitting can be observed even at the energy of 25 meV.

It should be noted that the data shown in Figure 5 are obtained after careful subtraction of the background intensity which was determined by the count rates of neighboring detectors of the PUMA multianalyzer system which are not used for polarization analysis.

Corresponding data obtained for the Brillouin zone (0 0 2) are shown in Figure 6a–f for energies close to the electromagnon excitations at 3 meV. There is a significant variation of the peak profiles: The NSF intensity profile is particularly broadened at 3 meV. This might be due to a coupling between the electromagnon and the phason mode. The SF data show the emerging peak of the tilt mode at energies above the spin gap of about 3 meV (cf. Figure 4c). Moreover, there is an increase in the diffuse intensity at lower energies which indicates either some disorder of the magnetic structure or just tails of the elastic satellite.

In the energy range between 12 and 14 meV (Figure 7), the shape of the NSF profile (Figure 7d–f) is altered just at 13 meV, the energy of the predicted electromagnon. Furthermore, the peak is broadened while at lower and higher energies, the profile is dominated by the bare magnon with its steep dispersion.

In the SF channel (Figure 7a–c), the profile variation is even more pronounced: While a single magnon peak is observed at 12 meV, there seems to evolve a side peak at 13, and at 14 meV, four individual components may be identified (blue lines in Figure 7c). In view of the low scattered intensity and the large error bars, the fitting procedure is, however, somewhat doubtful, but a single Gaussian fit (red line) leads to significant deviations. The almost symmetrical splitting of the beam profile might be the consequence of the beginning separation of magnon branches for positive and negative values of $\xi$. For the central doublet at $\xi \approx \pm 0.017$, the corresponding magnon stiffness $dE/dq$ can be roughly estimated as 400 meV Å$^{-1}$ if the spin gap of 3 meV is taken into account. This seems to be in fair agreement with the extrapolated value of Ain et al. obtained from high-energy data.$^{[15]}$

Additional data have been taken at low temperatures to obtain more information about the different magnon modes that have been postulated in the AF1 phase. Figure 8 shows the results of an energy scan at the magnetic zone center (2 0 2) + $q_C$ obtained at 5 K which may be compared with the data shown in Figure 5 of Jacobsen et al.$^{[20]}$ Due to the improved resolution of our own experiment using rather narrow collimation, the intensity is lower and the statistical error is considerably larger. But nevertheless, the well-defined minimum of the SF intensity near 19 meV is clearly reproduced. Moreover, there is evidence for some individual modes for energies near 3, 9, and 14 meV, in particular, which could not be resolved in the Jacobsen experiment. The present data are, however, not yet sufficient to improve the spin dynamics models of Gaw$^{[22]}$ or Jacobsen et al.$^{[20]}$

**4. Conclusion**

Polarized neutron scattering was used to characterize the fundamental magnetic excitations in multiferroic CuO. The new technique available at the thermal three-axes spectrometer PUMA@FRM II enabled us to detect intensities for SF and NSF channels simultaneously. Along with the choice of different Brillouin zones, different types of magnon branches could be identified along with their spin gaps at the magnetic zone center. There are significant differences between the low-temperature antiferromagnetic AF1 phase and the multiferroic AF2 phase which is characterized by a cycloidal magnetic structure. In
Figure 6. Constant energy scans for $E = 2, 3$, and 4 meV across the satellite position in the Brillouin zone (0 0 $\overline{2}$) of the AF2 phase at 218 K for a–c) SF scattering and d–f) NSF scattering. The lines represent fits using Gaussians.

Figure 7. Constant energy scans for $E = 12, 13$, and 14 meV across the satellite position in the Brillouin zone (0 0 $\overline{2}$) of the AF2 phase at 218 K for a–c) SF scattering and d–f) NSF scattering. The lines represent fits using Gaussians.
the AF1 phase, the modes with polarization along the easy-axis and the hard-axis exhibit a minimum energy of about 2 and 4 meV, respectively. Within the experimental resolution of about 1 meV, no energy gap is found for the phason mode of the AF2 phase. This is consistent with the fact that the cycloid has an almost circular shape. In contrast, the tilt mode is characterized by a well-defined energy gap of about 3 meV.

First data with improved resolution provide evidence of the existence of electromagnons as a magnon–phonon hybrid mode near 3 and 13 meV. Additional experiments are, however, needed to improve the statistics which are planned after the long shutdown period of the reactor.

5. Experimental Section

Elastic and inelastic neutron scattering experiments were performed using the three-axes spectrometer PUMA@FRM II[19] with its novel type of polarization analysis that allows the simultaneous detection of both spin states. While the incident neutron beam is polarized perpendicular to the scattering plane using a 3He-spin filter cell, the two spin states of the scattered neutron beam are spatially separated by FeSi multilayer deflectors and thus directed toward different channels (5, 6, and 7) of the PUMA multianalyzer. Details of this setup along with the method that allows the quantitative determination of SF and NSF intensities from the individual detector count rates are described in a previous study[19]. The basic relations include two general calibration parameters $D_+$ and $D_-$ as well as the polarization $P_0$ of the initial neutron beam provided by the 3He filter.

$$I_{SF} = \frac{1}{2} l_0 - \frac{D_+}{2} (l_3 + l_7) - \frac{1}{2P_0} (l_6 - D_+ (l_3 + l_7)) $$

$$I_{NSF} = \frac{1}{2} l_0 - \frac{D_-}{2} (l_3 + l_7) + \frac{1}{2P_0} (l_6 - D_- (l_3 + l_7)) $$

Due to the characteristic relaxation of the He polarization, $P_0$ is time dependent and has to be calculated for each individual scan point. The parameters $D_+$ and $D_-$ are determined to be 1.05 and −0.86, respectively.[19]

Neighboring multianalyzer channels are used to determine the instrumental background and allow the determination of background-corrected intensities.

It should be noted that the experimental uncertainties on the corrected intensities include a random contribution due to counting statistics as well as a systematic error due to the uncertainty in helium polarization. These errors are combined according to the usual covariance propagation law. The error bars shown in the figures, presented in this work, are the corresponding results which are dominated by the counting statistics of both signal and background channels.

The beam divergence was adapted by Soller collimators to 0.45° for the incident and 0.5° for the scattered beam. Guide fields between 3He filter and deflectors guarantee the conservation of neutron polarization. The initial polarization of the neutron beam was chosen to be about 90% and the 3He cells were exchanged after the polarization was reduced by about 15%. All data were taken with a constant final energy of 14.6 meV, and a PC filter was used to reduce the contamination of the detected beam by neutrons reflected in the higher order at the PC analyzer.

The sample of about 1 cm$^3$ volume was cut from a large boule obtained from MaTeck, Jülich, Germany, and characterized by gamma-ray diffraction. The overall mosaicity was thus reduced to 0.5°. The crystal was mounted in a closed-cycle cryostat with the monoclinic $b$-axis perpendicular to the scattering plane. The temperature was determined by a Pt100 sensor directly attached to the Al sample holder.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

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