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dr hab. inż. Piotr Konieczny, prof. IFJ PAN
The Henryk Niewodniczański
Institute of Nuclear Physics
Polish Academy of Sciences
ul. Radzikowskiego 152
31-342 Kraków

Report on doctoral dissertation of mgr **Mateusz Aleksander Gala** entitled:

Modification of quantum states in strongly correlated systems by applying uniaxial pressure

under supervision of:
dr hab. inż. Wojciech Tabiś, prof. AGH and Prof. Dr. Neven Barišić

General remarks

The doctoral dissertation under review, prepared in the form of a monograph and entitled "Modification of quantum states in strongly correlated systems by applying uniaxial pressure", concerns experimental investigations of the influence of uniaxial stress on the structural, magnetic, and electronic properties of materials exhibiting strong electronic correlations. The model system selected for this study is stoichiometric magnetite, Fe_3O_4, which, as convincingly demonstrated in the dissertation, despite decades of intensive research still remains a material with several unresolved and scientifically stimulating questions.

The main objective of the dissertation was "to investigate how uniaxial compression applied along the cubic [100] and [011] directions modifies the lattice symmetry of stoichiometric magnetite and how these structural distortions influence its electronic transport and magnetic response". This objective is well formulated, scientifically relevant, and embedded in the currently rapidly developing field of solid-state physics devoted to the use of pressure, both hydrostatic and uniaxial, as a controlled parameter for tuning electronic, magnetic, and structural properties in correlated materials.

To address the stated research problem, the Author employed a broad and appropriately selected set of advanced experimental techniques. These include structural studies by single-crystal X-ray diffraction (SCXRD), defect-field imaging using dark-field X-ray microscopy (DFXM), electronic transport measurements, AC magnetic susceptibility measurements, and X-ray absorption spectroscopy (XAS). Such a combination of complementary methods should be regarded as one of the strong points of the dissertation, since it allowed the Author to build a coherent picture of the coupling between lattice strain, electronic correlations, and the Verwey transition in magnetite.

The single-crystal X-ray diffraction experiments performed at the European Synchrotron Radiation Facility (ESRF) revealed a clearly anisotropic elastic response of magnetite to uniaxial compression applied along the [100] and [011] directions. Compression along [100] was shown to induce a tetragonal distortion, whereas compression along [011] led to an orthorhombic deformation. The studies of crystal defects using dark-field X-ray microscopy were also of considerable importance. They were used not only to investigate remanent strain fields persisting after decompression from the uniaxial pressure cell, but also to compare the microstructure of two types of high-quality magnetite single crystals: one grown by the travelling-solvent floating-zone method and the other obtained using the skull-melter technique.

The measurements of electrical resistivity and AC magnetic susceptibility under uniaxial compression along the [100] and [011] directions demonstrated an anisotropic enhancement of the Verwey transition temperature. This effect was interpreted in terms of distinct strain symmetries induced by compression (tetragonal for [100] and orthorhombic for [011]). This interpretation is physically well motivated and supported by a consistent analysis of the experimental results.

In my opinion, an equally important achievement of the dissertation is the part devoted to the design, construction, and calibration of a custom-built uniaxial pressure cell, referred to as the SOL cell. The Author presents an experimental device incorporating interesting technical solutions, including the use of a flat plate spring. Particularly valuable is the quantitative analysis of the applied stress performed using three independent approaches: analytical estimation based on the deformation of the flat plate spring and the simplified Euler-Bernoulli formalism, finite-element-method simulations, and experimental calibration by single-crystal X-ray diffraction. In addition, the Author constructed a dedicated setup for AC magnetic susceptibility measurements compatible with the developed uniaxial pressure cell. This part of the dissertation has significant value beyond the specific case of magnetite, since the developed pressure cell may serve as a versatile experimental platform for investigating the influence of uniaxial compression on a broad class of materials using various complementary techniques.

Overall, the dissertation is characterized by a well-chosen experimental strategy, high experimental competence, and a mature level of data analysis. The obtained results are original, scientifically significant, and of international relevance. They provide new insight into the anisotropic response of magnetite to uniaxial strain and contribute to the broader understanding of how symmetry-selective lattice distortions can be used to tune electronic and magnetic properties in strongly correlated systems.

Content of the thesis

The Author has carried out an ambitious and demanding research project, addressing an important problem in the field of solid-state physics. The dissertation not only demonstrates the richness of the collected experimental data, but also makes a significant methodological contribution through the development, calibration, and implementation of dedicated experimental instrumentation for studies under uniaxial pressure. This dual character of the work, which combines advanced materials physics with experimental technique development, should be regarded as one of its major strengths.

The dissertation is organized into six chapters, supplemented by six appendices, which constitute a valuable extension of the main text. The content of the dissertation is consistent with the aims declared by the Author, namely: (i) to investigate "how uniaxial compression applied along the cubic [100] and [011] directions modifies the lattice symmetry of stoichiometric magnetite and how these structural distortions influence its electronic transport and magnetic response, both at room temperature and in the vicinity of the Verwey transition" and (ii) to design, construct, and calibrate "a custom-built uniaxial pressure cell, specifically developed to perform the measurements presented in this dissertation".

Chapter 1 provides the conceptual and scientific background necessary for understanding the subject of the dissertation. It begins with a short introduction to strongly correlated electron systems. Then, the description focuses in more detail on magnetite, Fe_3O_4, which is the "main actor" of the dissertation. The discussion on magnetite covers its crystallographic structure, magnetic properties, electronic transport, and, most importantly, the Verwey transition. The Author presents the Verwey transition as a complex first-order transition involving magnetic, resistivity, and structural anomalies. The chapter also discusses the influence of non-stoichiometry, chemical doping, hydrostatic pressure, and uniaxial pressure on the Verwey transition, thereby clearly identifying the scientific gap addressed in the dissertation. The objectives of the work are formulated in a precise manner and naturally follow from this introductory analysis.

Chapter 2 is devoted to the experimental foundation of the dissertation and represents one of the most technically important parts of the work. The Author first reviews selected methods of applying pressure and then describes the construction of the custom-built SOL uniaxial pressure system. A particularly valuable part of this chapter is the discussion of the calibration strategy. The Author analyzes the applied stress using three complementary approaches: an analytical estimation based on the deformation of the flat plate spring and a simplified Euler-Bernoulli description, finite element method simulations, and an experimental calibration based on single-crystal X-ray diffraction. This multi-level calibration procedure significantly increases the reliability of the pressure scale used in the subsequent experiments. The chapter also contains a description of the synthesis of magnetite single crystals and the methodology of electrical resistivity and AC magnetic susceptibility measurements. The inclusion of the design of a miniaturized AC susceptibility setup compatible with the uniaxial pressure cell is especially noteworthy, since it shows that the Author did not merely adapt existing tools, but actively developed experimental capabilities required for the project.

Chapter 3 presents the structural response of magnetite to uniaxial compression. It begins with a brief introduction to the elements of elasticity theory and single-crystal diffraction. The experimental part is based on synchrotron single-crystal X-ray diffraction measurements performed under compression applied along selected crystallographic directions. The main result of this chapter is the demonstration of an anisotropic elastic response of magnetite. This is essential for the later interpretation of the transport and magnetic susceptibility measurements. The chapter also contains an important calibration of the SOL setup based on the evolution of lattice parameters measured under load by single-crystal X-ray diffraction. Therefore this part plays a central role in connecting the technical development of the pressure cell with the physical interpretation of the magnetite experiments.

Chapter 4 is devoted to the investigation of intrinsic and pressure-induced defect fields in magnetite using dark-field X-ray microscopy. The chapter first introduces the principles of DFXM and then applies this technique to compare the microstructure of high-quality magnetite single crystals obtained by different growth methods. This comparison is scientifically relevant, because defect structures, mosaicity, and axial strain may strongly influence the response of correlated materials to external stress. The Author demonstrates that the synthesis route leaves a clear microstructural fingerprint. This analysis justifies the selection of the most appropriate crystals for the subsequent uniaxial pressure experiments. The next section describe investigation which aim to determine how uniaxial stress modifies the defect field in stoichiometric magnetite single crystals and whether these stress-induced features persist in the relaxed state. In the case of [011] direction, extended dislocations persisted even after stress release and were clearly resolved in DFXM. In the context of Section 4.4, I would like to add one suggestion. It would be interesting to investigate, as an additional fourth case, the E_JD2 sample after several thermal cycles in which the sample is repeatedly cooled below the Verwey transition temperature, T_V. Such an experiment could provide valuable information on whether repeated crossing of the first-order Verwey transition affects mosaicity and axial strain, as probed by DFXM.

Chapter 5 presents the electrical resistivity, AC magnetic susceptibility, and local structural response of magnetite to uniaxial compression. It is one of the key chapters of the dissertation, as it directly addresses the central physical question of how direction-selective lattice distortion modifies the Verwey transition and the associated macroscopic properties. Electrical resistivity and AC susceptibility measurements revealed a strongly anisotropic enhancement of T_V under applied uniaxial stress. The importance of this result is further emphasized by the fact that these findings provide the first quantitative experimental verification of the anisotropic stress dependence predicted by Coe et al. The final part of the chapter is devoted to Fe K-edge X-ray absorption spectroscopy under uniaxial stress. The XAS measurements show no measurable changes in the pre-edge region within the investigated stress range, which suggests that that the local coordination symmetry around the Fe ions remains essentially preserved. In this context, EXAFS measurements under uniaxial stress would ideally complement the investigation presented in the dissertation, since they could provide direct information on stress-induced modifications of local bond lengths and coordination geometry. At the same time, I fully understand that, for a thick and iron-rich compound such as magnetite, self-absorption effects in fluorescence detection are severe and that reliable EXAFS measurements would be extremely difficult. Nevertheless, it would be interesting to know whether the Author has considered, as a possible future direction, EXAFS measurements using total electron yield (TEY) detection. Such an approach could reduce the limitations associated with fluorescence self-absorption, although it would also introduce constraints related to limited probing depth and increased surface sensitivity.

Chapter 6 summarizes the main results and conclusions of the dissertation. The Author brings together the structural, defect-related, transport, magnetic, and spectroscopic findings into a coherent physical picture. The main conclusion is that moderate uniaxial stress can tune the Verwey transition in stoichiometric magnetite in a strongly anisotropic manner. This chapter also emphasizes the broader relevance of the developed SOL pressure cell, which can serve as a general experimental platform for investigating the influence of uniaxial stress on a wider class of materials.

The dissertation is supplemented by six appendices, which provide useful technical and methodological details. They include information on sample preparation for resistivity measurements, construction of the miniaturized AC susceptibility setup, the finite element simulation environment, selected elements of elasticity theory, processing of DFXM data, and supplementary results of resistivity, susceptibility, and XAS measurements under uniaxial pressure. These appendices are not merely formal additions; they substantially improve the transparency and reproducibility of the work. They also demonstrate the Author's careful approach to experimental methodology, data treatment, and quantitative analysis.

Main achievements

The dissertation contains several important scientific and methodological achievements. The main scientific achievement is the demonstration that uniaxial compression can be used as a direction-selective tool to tune the structural, electronic, and magnetic properties of stoichiometric magnetite. The Author showed that stress applied along different crystallographic directions induces distortions of different symmetry. This structural anisotropy was convincingly linked with the pronounced anisotropic stress dependence of the Verwey transition temperature, observed independently in electrical resistivity and AC magnetic susceptibility measurements.

An equally important achievement is the design, construction, and calibration of the custom-built SOL uniaxial pressure cell. The developed device enables controlled and quantitatively calibrated uniaxial compression and was successfully implemented in several experimental techniques. Thus, the work contributes not only to the physics of magnetite, but also to the methodology of experimental studies under uniaxial stress.

Among other valuable achievements, I would also emphasize the comparison of structural imperfections in magnetite single crystals synthesized using the skull-melter and travelling-solvent floating-zone methods. Dark-field X-ray microscopy allowed the Author to reveal differences in mosaicity, axial strain, and extended defect structures.

I also appreciate the Author's awareness of the limitations of the applied methods, for example in the discussion of finite element simulations and the influence of sample mounting on the generated uniaxial force. This methodological caution strengthens the credibility of the analysis. The same applies to the careful description of electrical resistivity measurements under uniaxial compression.

A further noteworthy achievement is the development of the dedicated AC magnetic susceptibility setup compatible with the uniaxial pressure cell, described in Section 2.3.3 and Appendix B. This part clearly demonstrates the Author's high experimental competence. I only regret that Appendix B was not developed in greater detail, although I understand that not every technical aspect can be included in the dissertation.

Finally, the Author experimentally demonstrated that the SOL cell can be used for X-ray absorption spectroscopy under uniaxial stress. This confirms the versatility of the developed pressure platform and opens possibilities for future studies.

The scientific profile of the Author deserves a very positive assessment. He is a co-author of six publications in high-quality journals and has presented his results in nine oral contributions at conferences, workshops, and seminars, as well as in two poster presentations. Particularly noteworthy is his extensive experience in synchrotron experiments. The Author participated in eleven such experiments (eight at SOLARIS National Synchrotron Radiation Centre and three at European Synchrotron Radiation Facility - ESRF), including one at the ESRF in which he acted as the main proposer. He also completed three research stays at TU Wien and participated in four research projects, including one as principal investigator. The Author's academic activity also includes supervision of six students, three of them in connection with diploma theses, as well as substantial teaching experience. Together with scholarships and additional scientific activities, these achievements provide a strong basis for a high assessment of his scientific development and academic profile.

Criticism and questions

Although I assess the dissertation very highly, I would like to formulate a few critical remarks and questions, which are intended mainly as points for clarification and further discussion.

In some parts of the dissertation, a more explicit indication of the Author's individual contribution would be helpful. For example, in Section 2.1, Fig. 2.1b presents a prototype of a screw-driven uniaxial pressure system developed in collaboration between AGH and TU Wien. Since this setup is very similar to the SOL cell discussed later in the dissertation, I would appreciate a brief clarification of the Author's role in its design, construction, and further development.

The next comment concerns Section 2.1.2, which describes the construction of the SOL system. In my opinion, this part leaves some room for further technical detail. Since the SOL cell is one of the major methodological achievements of the dissertation, a more extensive description of its construction and mechanical limitations would have been valuable.

In pressure experiments, the determination of the initial measurement conditions is a key methodological issue. This is also relevant for the SOL system described in the dissertation. Therefore, I would like to ask how the Author determined the initial pressure in the SOL cells before starting the compression-dependent measurements.

In Section 2.1.6, the Author discusses the calibration of the SOL setup using single-crystal X-ray diffraction and the commercial ADMET pressure cell. The Author notes a difference between the calibration factor for SOL2 under axial compression along the [011] direction and SOL3 under compression along the [100] direction, with the former being almost twice as large. In my opinion, the explanation given in the dissertation is not fully satisfactory. A rough comparison of the ADMET and SOL data in Fig. 2.12 suggests that, for compression along [011], a stress of about 200 MPa in the ADMET setup corresponds to a screw rotation angle of approximately 180–210°. By contrast, in Fig. 2.13, for compression along [100], a stress of about 90 MPa corresponds to a screw rotation angle of approximately 150–200°.

I also noticed one conceptual simplification in the English abstract, where the Author writes that "hydrostatic pressure uniformly shortens all interatomic distances". Hydrostatic pressure is isotropic in terms of the applied stress, but the resulting structural response does not necessarily have to be isotropic, especially in anisotropic or low-symmetry materials. The same formulation appears in the German version of the abstract. The Polish version is slightly more neutral, stating that hydrostatic pressure systematically shortens all interatomic distances. Nevertheless, from a didactic point of view, such formulations should be used with caution.

Other, less significant remarks, for which I do not expect detailed answers during the defense, are listed below.

In a few places, the description lacks precision, which slightly hinders the reading of the dissertation. For example, in Fig. 1.3 on page 14, it is not stated whether the presented data were measured within the dissertation or taken from the literature; in the case of panel (a), the type of calorimetry used is also not specified. Similarly, in Fig. 1.10 on page 28, more information should be provided about the pressure cell used, the pressure-transmitting medium, and the method of pressure determination. The measurements were performed using a SQUID magnetometer, not vibrating sample magnetometry.

I also find the statement on page 151 somewhat too far-reaching: "Nevertheless, by analogy with resistivity experiments on both [100] and [011] oriented crystals, as well as susceptibility results under [011] compression, it can be reasonably inferred that the pressure-induced shift of TV is reversible also in the [100] orientation." In my opinion, such an inference should be formulated more cautiously, unless it is directly supported by experimental evidence for the [100] orientation.

There are also some minor editorial and graphical issues.

- In most figures the Author uses a comma as the decimal separator, but in several places a dot is used instead. It would be preferable to adopt one convention consistently, ideally using a dot throughout the English-language dissertation.
- Fig. 5.4 would be more informative if the data points corresponding to different samples were marked using different colors or symbols, as is done, for example, in Fig. 5.5.
- On page 153, in the last paragraph, the reference should be to Appendix F rather than Appendix A.
- On page 154, in the first paragraph, the reference to Fig. 5.14b should most likely be to Fig. 5.15b.

Concluding remarks

In conclusion, I assess the presented thesis very highly. The work addresses an important and timely problem in solid-state physics and presents original results of clear scientific value. I particularly appreciate the strong experimental character of the dissertation, including the development and calibration of dedicated instrumentation, as well as the broad and consistent use of complementary measurement techniques.

The critical remarks formulated above do not affect my very positive overall assessment of the dissertation. In my opinion, the thesis fully meets the requirements expected of a doctoral dissertation in the discipline of physical sciences.

Moreover, taking into account the very high scientific level of the dissertation, its important contribution to current research in solid-state physics, and the wide scope of the experimental work, I submit a motion to the Council of the Discipline of Physical Sciences of the AGH University of Krakow to distinguish the doctoral dissertation of Mateusz Aleksander Gala.

Summary in Polish

Podsumowując stwierdzam, że recenzowana rozprawa spełnia wymagania ustawy o stopniach naukowych i tytule naukowym oraz stopniach i tytule w zakresie sztuki określonej w art. 187 ustawy z dnia 20 lipca 2018 r. Prawo o szkolnictwie wyższym i nauce (z późn.zm.) i wnioskuję o jej dopuszczenie do dalszych etapów postępowania o nadanie stopnia doktora w dziedzinie nauk ścisłych i przyrodniczych w dyscyplinie nauki fizyczne.

Ponadto, mając na uwadze bardzo wysoki poziom naukowy rozprawy, jej istotny wkład w rozwój aktualnej problematyki fizyki ciała stałego, a także imponujący zakres przeprowadzonych badań eksperymentalnych obejmujących zarówno konstrukcję aparatury pomiarowej, jak i samodzielne projektowanie oraz realizację eksperymentów, zwracam się do Rady Dyscypliny Nauki Fizyczne Akademii Górniczo-Hutniczej z wnioskiem o wyróżnienie rozprawy doktorskiej Pana mgr. Mateusza Aleksandra Gala.

Piotr Konieczny

dr hab. inż. Piotr Konieczny, prof. IFJ PAN