MAcroscopic quantum Transport maTERials by nanoparticle processing

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MATTER

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Podsumowanie kontekstu i ogólnych celów projektu

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Nontrivial electronic band structures characterized by topological surface states in three-dimensional (3D) topological insulators (TIs) were experimentally demonstrated just over 10 years ago. Electrons on surface states are protected from backscattering, which means, they can move faster than electrons usually can. These fast-moving surface electrons make this material an exciting possibility

for electronics, and especially those that additionally take advantage of electron spin, known as spintronics. The problem, however, is that these materials - although called topological "insulators" - are still very good conductors of electric current in the bulk of the material. In fact, the electronic signal from within the materials dominates strongly. Therefore, unfortunately, it is still not possible to produce electronic devices that actually make use of this exciting possibility offered by the fast-moving surface electrons.
This is where the ERC Project MATTER comes in. Since the interior of the material is uninteresting for electronic applications such as spintronics and only the surfaces count, the approach of this project is to use nanoparticles. Nanoparticles, i.e. very small particles, have much more surface than any other representation of the material, e.g. thin films or tiny crystals. This allows one to emphasize the signatures of electrons on surface states in electrical transport. In this project, agglomerated nanoparticles - porous nanogranular solids - are used. But of course one encounters other challenges: the porous nanogranular solids are electrically difficult to contact and characterize, also the modeling of the data is certainly more difficult than for thin films, for example. The big advantage, however, is actually that the signatures of the surface states in the transport are strongly emphasized!

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The deeper understanding of electronically very special transport properties on surfaces of chalcogenide nanoparticles certainly has no one-to-one societal relevance in terms of direct applications. However, innovations in energy-saving technologies for data processing and storage have always emerged from basic research in the past. It is not at all foreseeable today what possibilities will arise from the use of electronic surface states, since even their characterization is still completely in its infancy. It is not even clear yet whether the applications really have to be in the field of electronics. Excellent conductivity on surfaces could also be interesting for energy-relevant applications such as catalysis. Therefore, a current focus of research is to establish the methods in both materials processing and measurement metrology to understand the transport properties of surface states as accurately as possible.

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In this project, nanoparticles are used to make the electronic transport properties of surface states experimentally accessible. Therefore, all four objectives aim at the synthesis, processing, characterization and utilization of these nanoparticles. The objectives are hereby to develop and optimize synthesis procedures for nanoparticles with the intended properties (objective 1), to fabricate suitable agglomerated nanoparticles - porous nanogranular solids (objective 2), to characterize thermoelectric and magneto transport of the porous nanogranular solids and to model the results (objective 3), and to test the obtained porous nanogranular solids in simple device structures towards their utilization (objective 4).

Prace wykonane od początku projektu do końca okresu sprawozdawczego oraz najważniejsze dotychczasowe rezultaty

Work performed in detail within the respective objectives:

Objective 1 (Nanoparticle synthesis) was done together with University of Duisburg-Essen, Prof. Stephan Schulz. The following nanoparticle samples were synthesized: 5g Sb_2Te_3, annealed at

400 °C; 5g Bi2Te3 with new thermally robust precursor, but not annealed; 4g of Te-enriched Bi2Te3 – another gram of this charge of material was directly delivered to a cooperation partner for THz spectroscopy, 5g of Bi2Te3, annealed at 300 °C, 5g of Bi2Te3 annealed at 400 °C, all samples including SEM, EDX, IR, XRD and most of them XPS analysis. Meanwhile, the first ternary samples were successfully synthesized: (SbxBi2-x)Te3 as well as Bi2(SexTe3-x). Additionally, also a larger amount of Bi2Te3 nanoparticles was produced with the intention to use this material as reference for additional studies on the processing parameters.

Objective 2 (Nanoparticle processing and interrupted early stage sintering) was developed by the implementation of a hot press together with a heating jacket into a glove box in order to provide a controlled experimental environment for the interrupted early stage sintering; Different sintering tools were tested, in part home-made by our workshop. The choice of the right sintering tool turned out to be extremely important to prevent pellets from cracking or even breaking when removed from the tool. Also, thin foils of high melting metals (Ta or W) were added between the pellets and the tools and thereby used to prevent the direct contact, the latter in order to avoid diffusion of Fe into the pellets. Sintering of test samples was done from the delivered nanoparticle batches; We optimized sintering temperature, time and pressure in order to control the porosity.
Owing to the fact that the start of the project was at IFW Dresden, one experimental study from this objective was continued there. This study dealt with Bi2Se3 nanoparticles obtained from ball milling and further compacted to pellets by spark plasma sintering. It could be shown that also these nanograined samples showed the signatures of a weak antilocalization connected with the existence of electrons in surface states. Still, the characteristic signatures of the weak antilocalization were weak compared to these signatures in Bi2Te3 nanoporous samples from the synthesis in ionic liquids combined with the mild hot pressing procedure.

In Objective 3 (Transport characterization), we designed and adapted several sample holders and measurement routines for two existing equipments such as a 4T cryogenics, as well as a Quantum Design MPMS measurement system. With that, high quality transport data could be acquired. Further, we analyzed all data by the Hikami–Larkin–Nagaoka model. For that, fitting routines were programmed. Also, the model needed to be developed slightly in order to account for the spin-orbit coupled transport. By doing so, we obtained exciting results, such as a coherence length of the spin-orbit coupled transport that is longer than the size of the nanoparticles.
Further spectroscopic characterization of the samples was done together with a collaboration partner, Prof. Martin Mittendorff (University of Duisburg-Essen). His team characterized samples by THz spectroscopy that were specifically produced for this purpose. By his characterization it could be shown that exciting properties of the surface states are not a low-temperature phenomenon. In contrast, a plasmon resonance closely connected with the high mobility of the electrons in surface states could be evidenced up to temperatures close to room temperature. Further, it could be modelled from his measurements that the electrons in the surface states dominate the transport properties at low frequencies.

For Objective 4 (towards devices), we developed a processing routine for topological insulator materials in thin films quality, as well as established microstructuring, so that the device fabrication can now start. The first devices will be thin film transistors (as a preliminary stage for spin devices), that are currently being fabricated. Our status of device fabrication is the following: we established a sputtering routine for SnTe films. We further established processing routines for two dielectrics, atomic layer deposition of Al2O3 as well as reactive sputtering of Si3N3, as well as for all the required metallization (Ta as adhesion layer and anti-diffusion layer, Au as contact layer). Currently our transistor devices are characterized by a too high leakage current through the gate dielectric. Hence, we now stepwise increase the thickness of the gate dielectric layer. We further already established an etching routine to artificially porosify the SnTe thin film and thereby increase the surface-to-volume ratio. Since atomic layer deposition conformly covers the surface of the underlying layer, these artificially porosified layers could then be used for devices as well. This work is ongoing and not yet at the level of publication.

Innowacyjność oraz oczekiwany potencjalny wpływ (w tym dotychczasowe znaczenie społeczno-gospodarcze i szersze implikacje społeczne projektu)

The most important and unexpected results that go far beyond the state of the art are:
• We have demonstrated very clear indications of spin-orbit coupled transport Bi2Te3 nanoparticular bulk samples that even resembles this of a superconducting transition. But it is clearly not a superconducting transition – this transition occurs when the surface states become dominant over the bulk carriers in the direct current electrical characterization.
• Additionally, it could be shown, for the first time, that the transport properties associated with the surface states are not a pure low-temperature phenomenon. With THz spectroscopy, resonance peaks could be assigned to the surface states, which could be seen in the experimental data almost up to room temperature. This robustness in terms of temperature stability was also not known before.
• When we further optimized the material, in particular, by the use of a slight Te excess, we could obtain the phase coherence length of the electrons on the surface states. For a highly porous sample, electrons on surface states had a phase coherence length of about 200 nm, which is significantly larger than the average size of the nanoparticles used – the latter was about 50 to 75 nm. Surprisingly, electrons on surface states can retain their coherence across nanoparticle boundaries. This kind of robustness was not experimentally evidenced before.

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