Report of my research stay at Purdue University in West Lafayette, USA

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From September to December 2018 I had the opportunity to visit the materials science and engineering department at Purdue University in West Lafayette, USA. I visited the group of Prof Edwin R. García, an expert in simulation and phase field modeling, and Prof. Haiyan Wang, who is an expert in TEM. The research stay was made possible by the funding of the Priority Program (SPP 1959), which I gratefully acknowledge. First, I'll talk about the scientific work and later one about my personal experiences abroad.

The focus of my SPP project is to investigate the influence of an external electric field on the grain growth in strontium titanate. I use dense sintered samples and current blocking electrodes to have a defined setup that allows to investigate only the effect of the external electric field on grain growth.

At Purdue I worked on two different projects. The first project was on flash sintering of doped strontium titanate, together with Prof. Wang. In the second project (with Prof. García) we worked on phase field modelling of the microstructure evolution in strontium titanate.

For the flash sintering project I prepared eight differently doped strontium titanate powders at Karlsruhe Institute of Technology (KIT) and brought them with me to the US. It was planned to conduct experiments on acceptor and donor doped strontium titanate, as the doping is changing the defect chemistry and the grain boundary potential. Previous experiments showed, that with increasing acceptor (iron) dopant concentration the conductivity of strontium titanate increases and thereby the onset temperature of flash sintering decreases. We were particularly interested in the grain boundary segregation behavior and the general grain boundary structure of the flash sintered samples, especially in the difference between the two electrode regions.

The flash experiments with donor doped strontium titanat resulted in samples with relative densities of ~80%. To improve the densification, all strontium titanate powders were remilled. By reducing the particle size by additional attrition milling, a higher relative density for all materials was achieved. Unfortunately, some samples showed contaminations that could be traced back to the milling balls used in Purdue at the end of my stay. Due to the contaminations, no TEM images were made. Back home, I send "clean" powder for Prof. Wang's group to repeat some experiments. Those sample are currently processed and investigated by means of TEM. Due to the incident with the powder, further TEM investigations were postponed.

The flash sintered samples prepared at Purdue showed, that the grains at the negative electrode region grow faster, as in experiments with an external electric field and the usage of current blocking electrodes. We further found out, that the microstructure at the positive electrode shows more pores and seems to be slightly less densified than the negative electrodes. The obtained mean grain sizes of the flashed samples at the negative and positive as well as at the sample center are shown in figure 1. The difference in grain sizes of the acceptor and donor doped samples can be attributed to the higher current limit used for the donor doped samples. A representative microstructure of a flash sintered sample, showing the difference in densification, can be found in Figure 2.

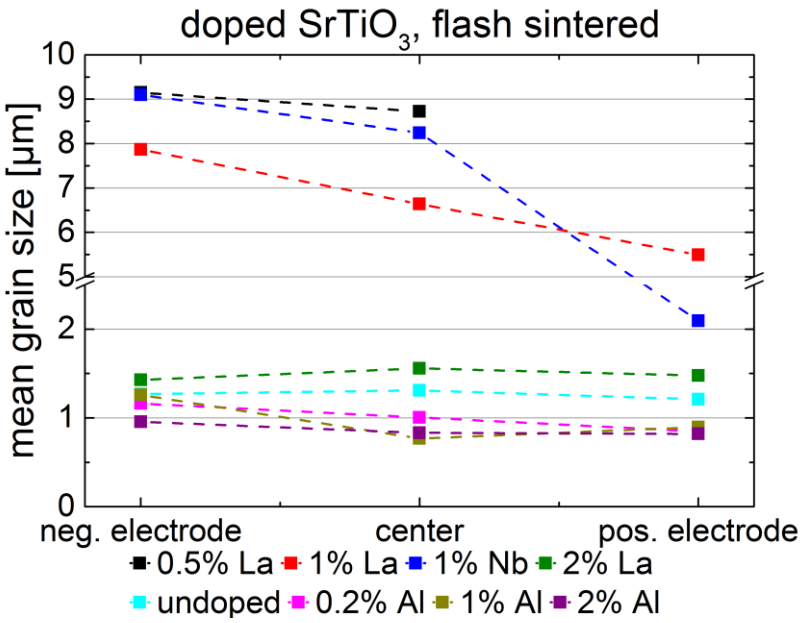

Figure 1: Mean grain sizes of the flash sintered samples, measured at the negative electrode, sample center and near the positive electrode. The value at the positive electrode for 0.5 % La-doped STO is missing due to local melting of the specimen

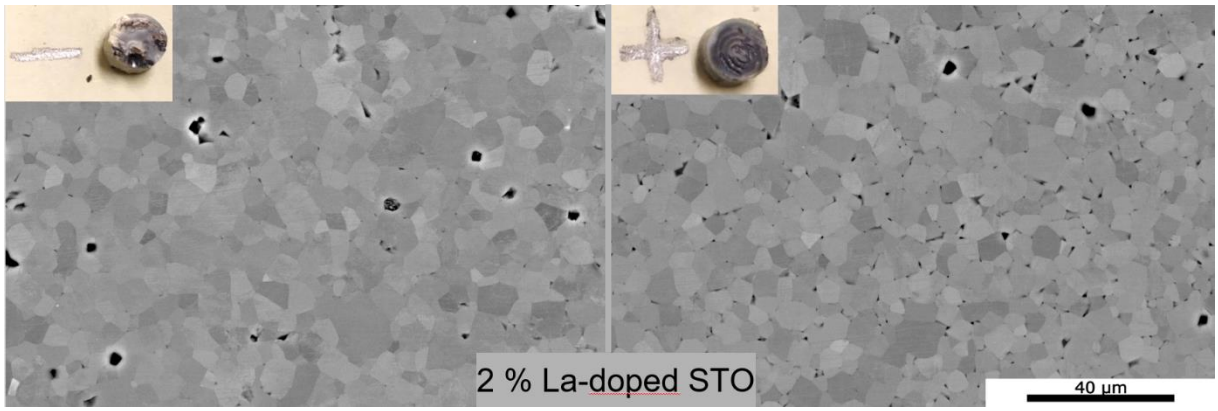

Figure 2: SEM images of the microstructure at the negative electrode (left) and at the positive electrode (right) of the 2% La-doped strontium titanate sample.

During the time I spent at Purdue, another group that is closely working together with Prof. Wang's group was interested in a collaboration with me. They do micromechanical testing on materials and asked for my flash sintered samples to make in-situ compression tests in the TEM. This collaboration is ongoing and results will be expected when the new "clean" samples will be completed by Prof. Wang's group.

In the second project at Purdue, I helped adapting a phase field simulation tool to strontium titanate. Literature and experimental data were collected and shared with Prof. García's group. The model was tested by using it to predict the spatial distribution of $[V_O^{\cdot\cdot}]$, $[V_{Sr}^{''}]$, $[h^{\cdot}]$, $[e']$, near a representative grain boundary of $SrTiO_3$. The obtained defect distributions are shown in Figure 3. So far the model predicted the existing data on strontium titanate correctly and will be further developed to simulate microstructural evolutions while changing certain parameters. Those results will soon be published. Figure 3 shows that lower angle grain boundaries exhibit Debye-type behavior and higher angle grain boundaries follow Mott-Schottky type behavior.

The group of Prof. García is further developing models to predict the grain boundary potential and the adjacent space charge layer by incorporating chemo-mechanical components in the code. These models are planned to be used for the simulations on strontium titanate microstructure evolution as well and to extract the grain boundary mobility with an applied electric field. We will continue our collaboration further as I acquire more experimental data.

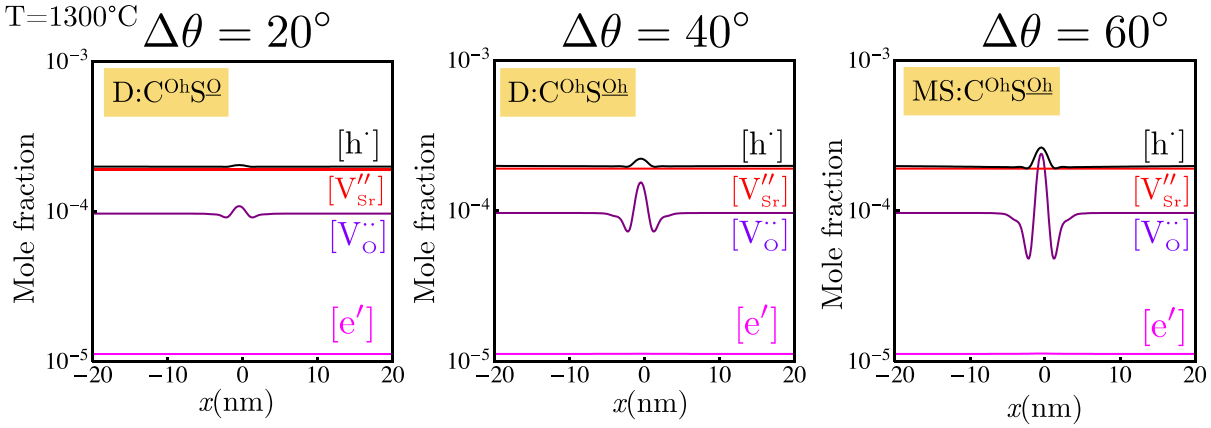

Figure 3: Simulated defect distribution in the vicinity of grain boundary for selected misorientations, 20°, 40°, and 60° at T = 1300 °C. 20° and 40° exhibit Debye-type space charge layers and 60° is reminiscent to Mott-Schottky type space charge layers.

For example, investigations of the effect of different oxygen partial pressures and electric fields on the grain growth are planned.

Besides the scientific work, I would like to share some personal experiences I made in the US and in particular at Purdue University. When I arrived, I went to the international office and they helped me with getting a student ID, access to the IT system and things like that. At the material science and engineering department I got a desk in a grad student office with a key for it. Everything was relatively uncomplicated, as the American people are very open, friendly and service oriented. To achieve an access to the labs was a bit more difficult, as the labs have electronic locks with personalized access regulations. This is done because the building was always open and the personalized access was also used to make sure that a lab usage fee was payed. Most labs at MSE in Purdue were shared because undergraduate students and grad students of all groups jointly used the labs. Unfortunately, these jointly used labs were not so clean and well-ordered as the labs at KIT and working in these labs was often challenging. The introduction to the devices in the labs was made by fellow grad students without any problems. Most of the grad students have multiple projects they are working on besides attending lectures. Which means that the whole week (including weekend and the night) is used to mix studying, research, sports and free time. Some students worked primarily from home and went only to the university for lectures or meetings. In contrast to the German system, where the group has often lunch together at the Mensa, there is no Mensa in the US. There was further no coffee and tea break where, at least in Germany, a lot of informal discussion happens.
The university is more like a little town within a town and the students were heavily involved in all kinds of different clubs, sports teams and other activities. The football matches of the university team were a highlight where everyone joined the party and parts of the campus turned into a festival like zone. As sports is a major part of campus life, the student's gym was the largest and best equipped gym I've seen so far.
Getting a room for my stay was challenging. I started at the beginning of the semester and most places only offer 12-month lease contracts. I found a nice place at craigslist.com, something similar as ebay-kleinanzeigen. I used a standard lease contract and payed the rent in cash. This is the easiest way, as bank transfers are not as common in the US as here and I needed a contract to get reimbursement for the rent by the SPP.
During my stay I also visited Chicago and drove around Lake Michigan. Thereby I experienced the American people and the American culture in many different aspects besides the American university culture. This was very valuable as it helped me to understand the American culture better.

This report could not cover all the things I experienced at Purdue, at the US and by organizing this stay, so feel free to ask me further questions! I am very happy to help you out (jan.preusker@kit.edu)