Österreichische Keramische Gesellschaft

Erste Mitgliederversammlung
7. Februar 2013

Seminarraum F (EG Hörsaalgebäude)
Montanuniversität Leoben

Franz-Josef-Straße 18
A-8700 Leoben

T A G E S O R D N U N G

9:30 Registrierung

10:15 Begrüßung

10:20 Präsentationswettbewerb (Teil I)

12:00 Mittagsbuffet

13:30 Präsentationswettbewerb (Teil II)

15:10 Kaffeepause

15:30 Mitgliederversammlung

- Bericht des Obmanns und des Kassiers
- Bestellung der Rechnungsprüfer
- Wahl des Vorstandes
- Diskussion über Schwerpunkte und Entwicklungen auf dem Gebiet der Keramik in Österreich
- Vorschläge über Fördermaßnahmen der Österreichischen Keramischen Gesellschaft
- Vorschläge zu Preisen und Ehrungen
- Allfälliges

17:30 Preisverleihung an die Gewinner des Präsentationswettbewerbs

18:00 Informelle Nachsitzung (Weinlaube Schwarzer Hund)

Präsentationenliste

	WettbewerberIn	Titel	Startzeit
DI	Magdalena Drózd-Ryś	Carbon burnout in magnesia-carbon refractories during ladle preheating	10:20
DI	Christoph Fasching	Simulation of crack formation during cooling of a MgO/MA-spinel refractory	10:40
DI	Michael Hofstätter	3D network-based simulation of the piezoelectric behavior of varistors	11:00
Dr.	Shengli Jin	Determination of Young's Modulus, Fracture Energy and Tensile Strength of Refractories by Inverse Estimation of a Wedge Splitting Procedure	11:20
DI	Irmtraud Marschall	Melting behaviour of mould powders for the continuous casting process of steel	11:40
DI	Gerald Mitteramskogler	Additive Manufacturing of parts made of Tricalcium Phosphate	13:30
DI(FH)	Sören Röhrig	Self-regulating $BaTiO_3$-based PTC heaters with interdigital electrode design	13:50
BSc	Peter Schneider	Process parameter optimization and filler surface modification in SiC-filled polysiloxane-derived ceramics	14:10
Mag.	Denis Schütz	Covalence in Ferroelectric Oxides – From the theory of relativity to industrial application	14:30
DI	Stefan Strobl	The effect of crack healing of silicon nitride evaluated with the "Notched Ball Test"	14:50

Abstracts

Carbon burnout in magnesia-carbon refractories during ladle preheating

Magdalena Drózd-Ryś

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Magnesia-carbon refractories, which are applied in the steel ladles in slagline area, have high chemical, mechanical and thermal resistance, but one of their major disadvantages is the possibility of carbon burnout. This may occur during preheating of the steel ladle after refractory relining. To reduce the thermo-shock of the refractories at the start up, the steel ladle is preheated up to 1100°C with natural gas firing with a significant amount of excess air. The preheating conditions in the ladle favor the carbon burnout in magnesia-carbon refractories.

The objective of the project is to examine the impact of carbon burnout on the wear of magnesia-carbon refractories and in consequence on the ladle lining lifetime. The approach for this investigation is to define the reaction rate of carbon oxidation to carbon monoxide.

The reaction kinetics depends on mass transfer coefficient, which defines oxygen transport onto the refractory surface, diffusion coefficient, which defines oxygen transport in void space of refractory and reaction rate coefficient, which defines the speed of chemical reaction.

The reaction rate coefficient of carbon oxidation is assumed to have no considerable influence on the reaction.

The diffusion coefficients were determined experimentally using the thermogravimetric analysis. As the diffusion coefficients showed dependence on the temperature, the results of the investigation could be presented in form of Arrhenius equation. The other influence factor was the carbon content of the refractory.

The mass transfer coefficient will be determined with help of computational fluid dynamic simulations. For that purpose, a commercial program FLUENT was applied to calculate gas and refractory temperature distribution as well as oxygen concentration in the ladle during its preheating. For the gas combustion a non-premixed combustion model including steady laminar flamelet approach was applied. The radiation is calculated with discrete ordinates model, the turbulence with realizable k-ε model. The calculation's results deliver the necessary data to define mass transfer coefficient and in further step the reaction rate of carbon burnout. To evaluate the simulation results laboratory scale experiments are planned in the future.

Simulation of crack formation during cooling of a MgO/MA-spinel refractory

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This presentation is about the simulation of crack initiation and propagation in a refractory structure during the cooling stage of the firing production. The mechanical behavior of refractories is mainly defined by the structure and its constituents and cannot be calculated in a straightforward manner. For example the Young's modulus of a MgO/MA-spinel refractory material may be 80 GPa although the grains have Young's moduli of approximately 300 GPa and the uncracked matrix has a Young's modulus of 110 GPa. The impact of parameters like the grain size distribution, the volume fraction, the thermal expansion mismatch and others on the fracture during the cooling process will be quantified with FE-simulations. For this approach a two dimensional representative volume element (RVE) with periodic boundary conditions and randomly distributed grains will be generated. The observed initiation and propagation of cracks during the cooling stage of the firing process is simulated with the commercial software ABAQUS. Afterwards the tensile strength and the fracture energy at room temperature are determined in a subsequently simulated tensile test.

3D network-based simulation of the piezoelectric behavior of varistors

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Varistors (i.e. VARIable resiSTORs) are electroceramic devices for over-voltage protection. In normal use the varistor is highly resistive, beccause of a negative sheet charge in the grain boundaries, the so-called double Schottky barrier, acting as a potential barrier for the electrons. Above a certain voltage, the breakdown-voltage V_B, electron-holes are generated by impact ionization, which compensate the negative sheet charge and the electric barrier is lowered within a small voltage range. This leads to a highly non-linear voltage-current characteristics, where the current I is proportional to the voltage V^a, with a-values up to 200.

Measuring the electric properties of varistors under varying mechanical loading conditions a strong pressure dependence of the I-V characteristic can be shown. The leakage current increases more than 2 order of magnitudes at 200 MPa pressure. This electro-mechanical behavior is explained by piezoelectric induced surface charges, which modify the sheet charge layer in the grain boundary and shift the height of the potential barrier up or down depending on the polarization vectors. This leads to an increase and decrease of the resistance of the single current paths through the varistor. But the device resistance is determined by the current path with the lowest resistance and so the leakage current increases.

A 3D network-based model of the varistor will be presented to analyze this physical effect. In this model each grain boundary is represented by a non-linear resistor, which characteristic is determined by the local microscopic properties, like the polarization vector of the adjacent grains. The polarization vectors are obtained by a mechanical simulation with Finite Element Method with a realistic microstructure, where the externally induced stresses due to the orthotropic elastic behavior are calculated. Also the residual stresses due to the cooling after the sintering process and an anisotropic thermal expansion coefficient are considered. A comparison of the simulations and measurements will be shown.

Determination of Young's Modulus, Fracture Energy and Tensile Strength of Refractories by Inverse Estimation of a Wedge Splitting Procedure

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Ordinary ceramic refractory materials generally possess a comparatively complex microstructure, which consist of grains in wide size distribution, matrix, and large defects e.g. pores and cracks. Consequently, the mechanical fracture behaviour of these materials profoundly deviates from pure linear elastic fracture mechanics (LEFM). In case the crack initiates or propagates a process zone occurs in front of and behind the crack tip. Mechanical phenomena, for instance, crack branching, grain bridging, friction of crack faces, or plastic behaviour at high temperatures could contribute to the deviations from LEFM. For simplification, fictitious crack model (FCM) was proposed by Hillerborg especially dealing with Mode I failure. Based on the theory of FCM, the stresses after cracking decrease with respect to crack opening till the opening reaches the ultimate allowed displacement, and thereby fracture energy can be calculated. Wedge splitting test is well applied in the refractories field to utilize the fracture energy concept and recently draws much attentions of refractory scientists, thanks to the guarantee of stable crack propagation. However, by this method only nominal tensile strength and specified fracture energy can be directly calculated from the measured force-displacement curve.

In order to obtain the pure tensile strength, theoretical fracture energy or Young's modulus from the wedge splitting test, a general inverse estimation procedure is developed in the frame of ABAQUS and the open source Dakota. The local minimization is realized by NL2SOL in which an adaptive choice of two Hessian models is implemented and consequently a fast convergence from poor starting guesses can be promoted. The sensitivity among the number and values of parameters of FCM are investigated and the capacity of the inverse estimation procedure as well. The results exhibit the strong robustness and high efficiency of this inverse estimation procedure. A trilinear strain-softening law provides excellent predictive accuracy, as well as bilinear strain-softening law in the case of determined Young's modulus. Finally, the inversely estimated parameters from three experimental curves are shown and compared as well as the values of R and l_{ch}.

Melting behaviour of mould powders for the continuous casting process of steel

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Mould powders are additives in the continuous casting process. They are added to the meniscus within the mould. Due to the high temperatures it melts and infiltrates the gap between the mould and the strand to lubricate the strand and control the heat flow. One of the most essential parameters in the continuous casting process to provide casting conditions stability and product surface quality is the melting behaviour of the mould powder. Operating experience has shown that the knowledge of only the chemical composition is not always sufficient for the mould powder selection. It is likely that beside the chemical composition the phase composition and its distribution are of influence on the mould powder performance.

Therefore the phase compositions of fourteen commercial mould powders were investigated by reflected light and scanning electron microscopy as well as X-ray diffraction. Several methods have been applied to investigate the melting behaviour of these powders. After annealing at different temperatures specimens have been investigated by X-ray diffraction, reflected light microscopy, scanning electron microscopy and X-ray microanalysis. Furthermore simultaneous thermal analysis and hot stage microscopy have been applied. The CaO/SiO_2 ratio of the specimens was in the range of 0.6-1.3 and the carbon content between 2.2% and 15.2%. Although the mineralogical composition of the mould powders as received differs, there are still similarities concerning their components.

The analysis of the green mould powders shows that the biggest part of the phases are silicates. Partially synthetic glasses like glass scrap or blast furnace slag are one of the main phases. Fluorite (CaF_2) is always used as a fluorine source. Sometimes cryolithe (Na_3AlF_6), phosphoric slag and villiaumite (NaF) are utilised as well. Sodium carbonate (Na_2CO_3) is added as sodium carrier, but in some cases during the granulation process be-carbonates e.g. gaylussite ($Na_2Ca(CO_3)_2$ are formed as a result of the reaction between sodium carbonate and lime silicates. Important components are carbon carriers. In minor amounts fused alumina (Al_2O_3), calcite ($CaCO_3$) and magnesite ($MgCO_3$) are present. As a lithium source lithium carbonate (Li_2CO_3) and spodumen ($LiAlSi_2O_6$) is used.

According the melting behaviour the mould powders show some similarities, too. In all cases the carbonates start to dissociate below 500 °C and finish not later than 790 °C. As a consequence Na_2O diffuses into the surrounding silicates and lowers their melting range. The formation of the first liquid phases depends on the used silicates. Especially the presence of glass scrap leads to the formation of liquid phases at low temperatures and enhance the melting of mould powders. Although the investigations were performed with a melting rate of 5K/min the time was too

short to reach thermodynamic equilibrium. As a consequence especially results from hot stage microscopy were influenced by the presence of carbon carries, because carbon is non-wettable by silica melts and hinders melt droplets from agglomeration. Between 750°C and 900°C sodium-lime-silicates recrystallize out of the sodium saturated liquid. Due to the dissolution of Alumina and silicates containing Al_2O_3 > 18 mass% sodium-alumina-silicates are formed between 900 °C and 1000 °C. Cuspidin is the most important phase formed within fluorine containing silica based mould powders because it is the longest lasting solid phase during melting. At temperatures around 600 °C it is formed due to diffusion of fluorine into sodium silicates. But the main rate recrystallizes out of a liquid phase between 690 °C and 1073 °C when a considerable amount of liquid is present. By contrast a not otherwise specified phase containing 17,1 mol% Al was formed in the only calcium alumina based mould powder investigated above 900 °C due to its high alumina content of 25,3 mass%. In this case cuspidine was no longer the main crystalline phase. The formation of a homogeneous liquid phase takes place between 1020 °C and 1260 °C and depends on the CaO/SiO_2 ratio of the mould powders.

Additive Manufacturing of parts made of Tricalcium Phosphate

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For their application in medicine, biodegradable ceramics offer many advantages. Unlike metallic materials, they are radiolucent and may degrade within the body in a period of 6-12 months. Due to their osteoinductive and osteoconductive properties they are also interesting for the use as scaffold materials for regenerative medicine. This new tissue engineering approach requires a scaffold with defined porous architecture and predictable physical properties. Providing a specific architecture requires innovative manufacturing techniques. In the scope of this work, a lithography-based additive manufacturing technique was investigated for its suitability of structuring tricalciumphosphate.

Additive manufacturing Technologies (AMT) are moving more and more away from the fabrication of prototypes to the manufacturing of functional components. AMTs are used for the shaping of geometrically demanding ceramic materials in a small batch series. At the Vienna University of Technology a novel AMT system („Blueprinter)“ was developed specifically for this purpose. The flexibility of the system and the achieved product properties legitimize its use over conventional methods.

The objectives of this work are the variation of the used photoreactive slurry and the optimization of the manufacturing process parameters for the flawless production of dense ceramic structures with good precision and high surface quality. The first part of this work deals with the ideal mixture of substances, namely the composition of the TCP powder and photosensitive resin. The interaction of the used components and their effect on the slurry properties (rheology, reactivity, processability) and the final product (surface quality, cracks) are examined in the second part. Finally, the end product is characterized in terms of mechanical and microstructural properties.

Self-regulating $BaTiO_3$-based PTC heaters with interdigital electrode design

Sören Röhrig[1] and Peter Supancic[1,2]

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Barium titanate based PTC-material shows an exponential increase of the resistivity within small temperature range related to the ferroelectric-paraelectric phase transition (PTC effect), which occurs at the so-called Curie temperature. This behaviour is used in several switching and sensor applications, among them self-regulating heating elements (PTC heaters).

Conventional PTC-heaters are simple cuboids or disks, whereby the electrodes for the electric excitation were located on two opposite surfaces of the device. This established design has some disadvantages with respect to applications with high voltage and high heat output, respectively. On the one hand, a thin geometry is necessary for a good heat performance, on the other hand a minimum electrode distance must be maintained.

In this work an alternative electrical excitation for PTC heaters is investigated, which is based on the well-known interdigital electrode (IDE) design. This electrode design is located on the same surface and overcomes the mentioned drawbacks, because electrode distance and thickness of the device can be adjusted independent to each other, in order to find a respective optimum.

Investigations on the underlying mathematical differential equation in a dimensionless form provide an efficient way to analyze both designs of the heater in order to outline the different electrothermal behaviour. In certain cases by the alternative IDE design the heat output into a medium is increased by a factor of two and more, while the switching behaviours is smoother.

Experimental investigations based on electrical and thermographical measurements on PTC heaters with IDE design were compared to various formed theoretical models (*i.e.* 1-, 2-, 3- dimensional). Features of the IDE design, which were predicted with these models, were also observed with lock-in thermography.

In further works the dynamic switching behaviour which occurs in certain cases has to be investigated in detail. Another challenge will be to find an appropriate material-combination for mechanical support. Thus has to provide a good thermal conductivity as well as an electric insulation.

Process parameter optimization and filler surface modification in SiC-filled polysiloxane-derived ceramics

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Manufacturing bulk ceramics from polymeric precursors is an interesting alternative to conventional ceramic production routes. Si-based preceramic polymers are converted into ceramic materials during a thermal (pyrolytic) conversion step. The main advantage of the precursor route is the fact that resilient green bodys can be obtained. This enables complex shaping of the ceramic product in its green state, resulting in higher cost-efficiency during machining. The main disadvantage of the polymer route is the high shrinkage during pyrolysis as a result of mass loss as well as an increase in density of the constitutents. To overcome this challenge, filler particles are generally introduced into the preceramic polymer matrix. Inert and reactive filler particles can be used. In this work the main focus of attention is on inert filler particles, more precisely silicon carbide (SiC).

The goal of this work is the identification of relevant process parameters during the fabrication of polymer-derived ceramics with SiC filler particles. Furthermore, the involvement of the filler particle surface on resulting structural and mechanical properties of the composite shall be investigated. Samples were produced by warm-pressing of the polymer-filler mixtures with a subsequent thermal conversion step. Three main process parameters of the warm-pressing method were investigated: the polymer volume content of the mixture, the pressure during warm-pressing, and the particle size of the silicone carbide filler particles. An assessment of the respective influence of each parameter was obtained by a multi-dimensional experimental design approach (“design of experiments”, DoE), using the “Essential Experimental Design” software package. The resulting composite’s density was chosen as characteristic output parameter. Concerning the compaction of the composite mixures during warm-pressing, it was shown that the particle size plays an important role. Particle sizes below 1 µm turned out to be detrimental to the compaction of the specimens. Green densities around 96 % could be obtained by using coarse fillers, high polymer volume contents, and medium compaction pressures.

In the second part of the investigations, the involvement of the filler particle surface on the resulting properties was evaluated by the application of different types of surface treatment to the filler particles before incorporation into the polymer matrix. Surface alteration included functionalization by several types of coupling agents (silanes and organic acids) as well as thermal treatments. Process parameters during the following specimen production steps were derived from the optimized production approach obtained by experimental design. The produced composites were compared with respect to their density, hardness, flexural strength, and Young’s modulus, demonstrating the influence of the filler particle surface constitution on the resulting composite’s properties.

Covalence in Ferroelectric Oxides – From the theory of relativity to industrial application

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The enactment of the two pieces of European legislation, Reach and RohS, and the subsequent prohibition of the use of lead in electrical components, has put a tremendous challenge to the industry as well as scientists working on ferroelectrics. While exemptions exist for crucial applications while no other technology is viable, the most widely used actuator material Lead Zirconium Titanate-PZT (and indeed most industry standard ferroelectrics) will be phased out in the next decade. Bismuth Sodium Titanate has emerged as the frontrunner to replace lead based compositions; however its excellent but unusual thermal and field dependent properties have been poorly understood so far.

In this talk I will illustrate the reasons why lead is so hard to replace in ferroelectrics, specifically actuators. This will start by illustrating the relativistic quantum chemistry of heavy cations like lead, thallium and bismuth and how this, via their inert lone pairs leads to an intracellular bonding not unlike a chemical complex. This in turn leads to explain how the thus induced covalence leads to the peculiar phase transitions and field induced strain that allows Bismuth Sodium Titanate to be superior to PZT in most respects relevant to actuators. As the measurements were carried out on our prototype multilayer, using industry standard, water based techniques for manufacture, they will also show that theory needs not be far from application, but can be applied side by side for the benefit of both.

The effect of crack healing of silicon nitride evaluated with the “Notched Ball Test”

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In the last decades ceramic components were more and more used in high performance hybrid bearings. The use of structural ceramics due to their special properties (low wear rates, low density, high corrosion resistance) is advantageous for applications requiring high operation speeds, electrical insulation (e.g. in windmills), or which suffer from severe corrosive attack (e.g. chemical or food industry).

The strength of ceramic rolling elements (relevant for construction and lifetime prediction) depends on the quality of their original surface, which may also depend on machining and handling. Although information on the strength of rolling elements is essential, simple strength testing methods of the original components are missing up to now.

A new testing method for ceramic balls – the Notched Ball Test – is presented to determine the surface strength. By this simple procedure the original surface of large samples can be tested in terms of the production process or special defect types, e.g. cracks or grinding scratches. The influence of artificial cracks on fracture stress is evaluated (compared with the strength of undamaged components) and - in addition - the effect of crack healing via heat treatment of the silicon nitride balls is presented.

It can be shown that the value of the resulting fracture stress of the damaged and healed components can be increased by about 100% and reaches nearby the original strength of undamaged bearing balls.

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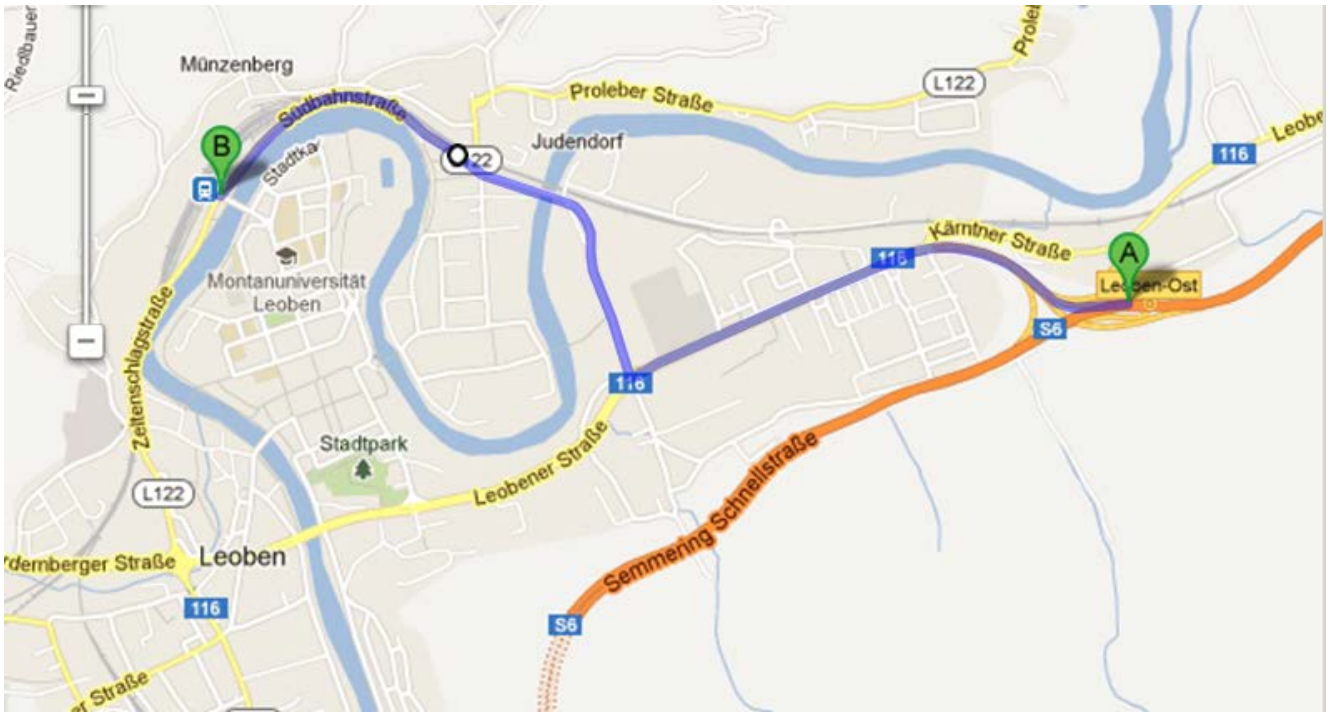
Anfahrtsplan

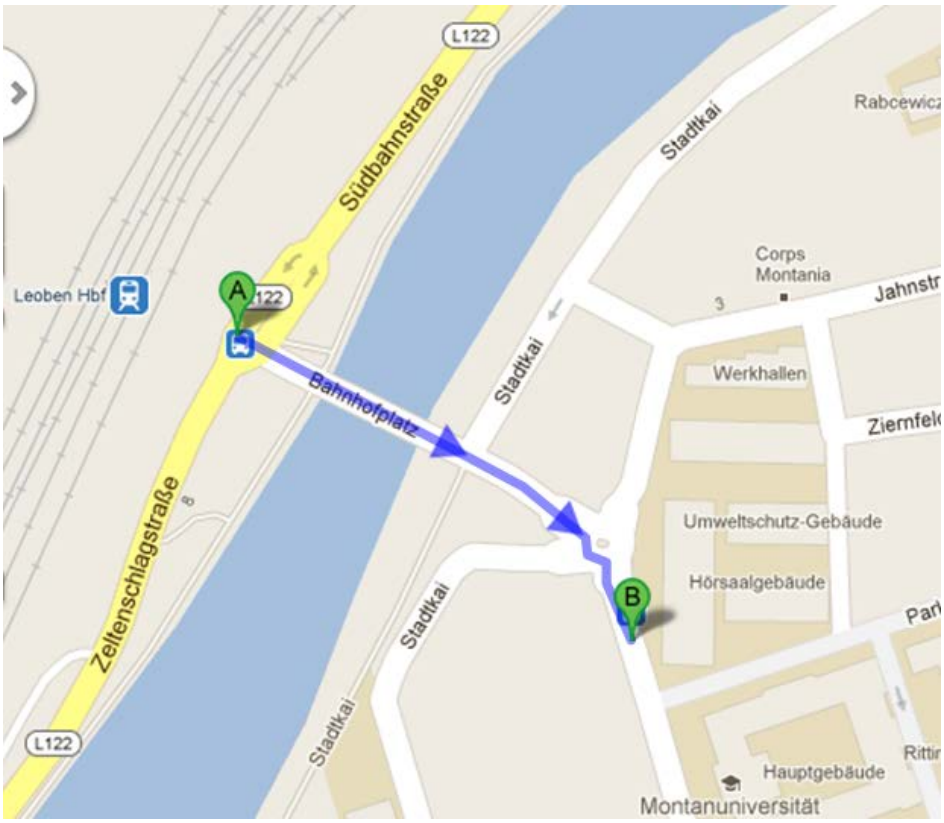
A – Leoben Ost

B – Hauptbahnhof



A – Hauptbahnhof

B – Montanuniversität Leoben



Parkplätze sind auf dem Parkbereich A der Montanuniversität verfügbar.