RUHR-UNIVERSITÄT BOCHUM

RUB

MATERIALS RESEARCH DEPARTMENT

FOCUS ON:

↗ **Sustainable Materials**

NO. 14

Materials Research Department

MRD
MATERIALS RESEARCH
DEPARTMENT

Welcome to the 14th MRD newsletter and its new look! With this new design, we join a lot of new beginnings for our Materials Research Department in 2022: Anjana Devi has taken office as the first female co-speaker of the MRD. In May, we not only held our MRD Industry Day in-person for the first time, but also celebrated the official opening ceremony of the research centre ZGH (Center for Interface-Dominated High Performance Materials).

FOCUS TOPIC: SUSTAINABLE MATERIALS

New beginnings and further investigations are also much needed in this topic: Sustainable materials. New materials and processes in all areas of materials science are required to enable future technological developments in areas such as sustainable energy technologies and energy-efficient processes. Our 14th MRD newsletter gives insights into the research activities on sustainable materials and processes within the MRD.

Like all other previous newsletters, this 14th issue is also available online on the MRD website. We look forward to your feedback and welcome suggestions for new focus topics for the coming issues of the MRD newsletter.

Enjoy reading,

R. Drautz and A. Devi
MRD Speakers
P. Aleithe and F. Scholz
MRD Science Managers

CONTENT

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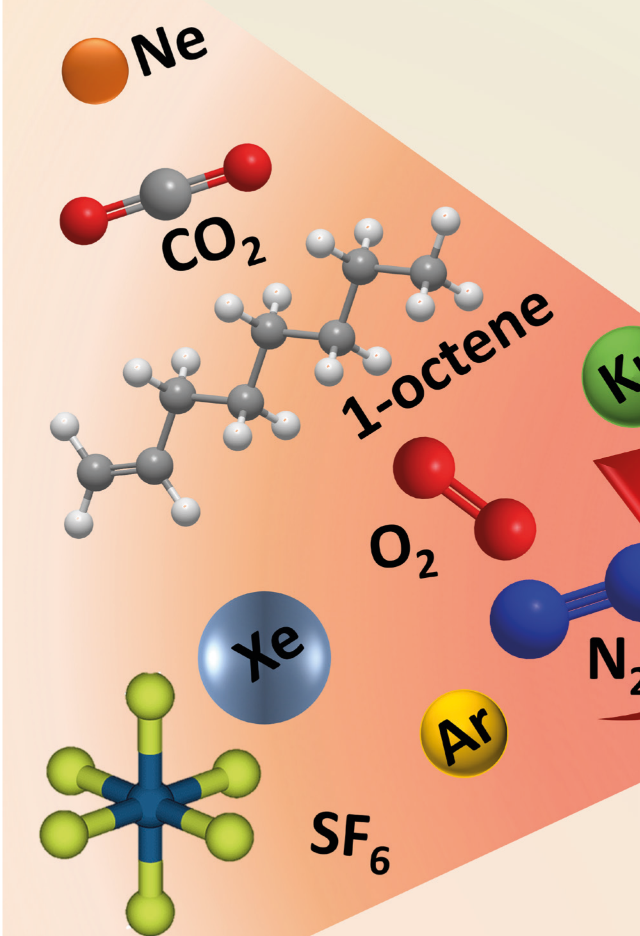
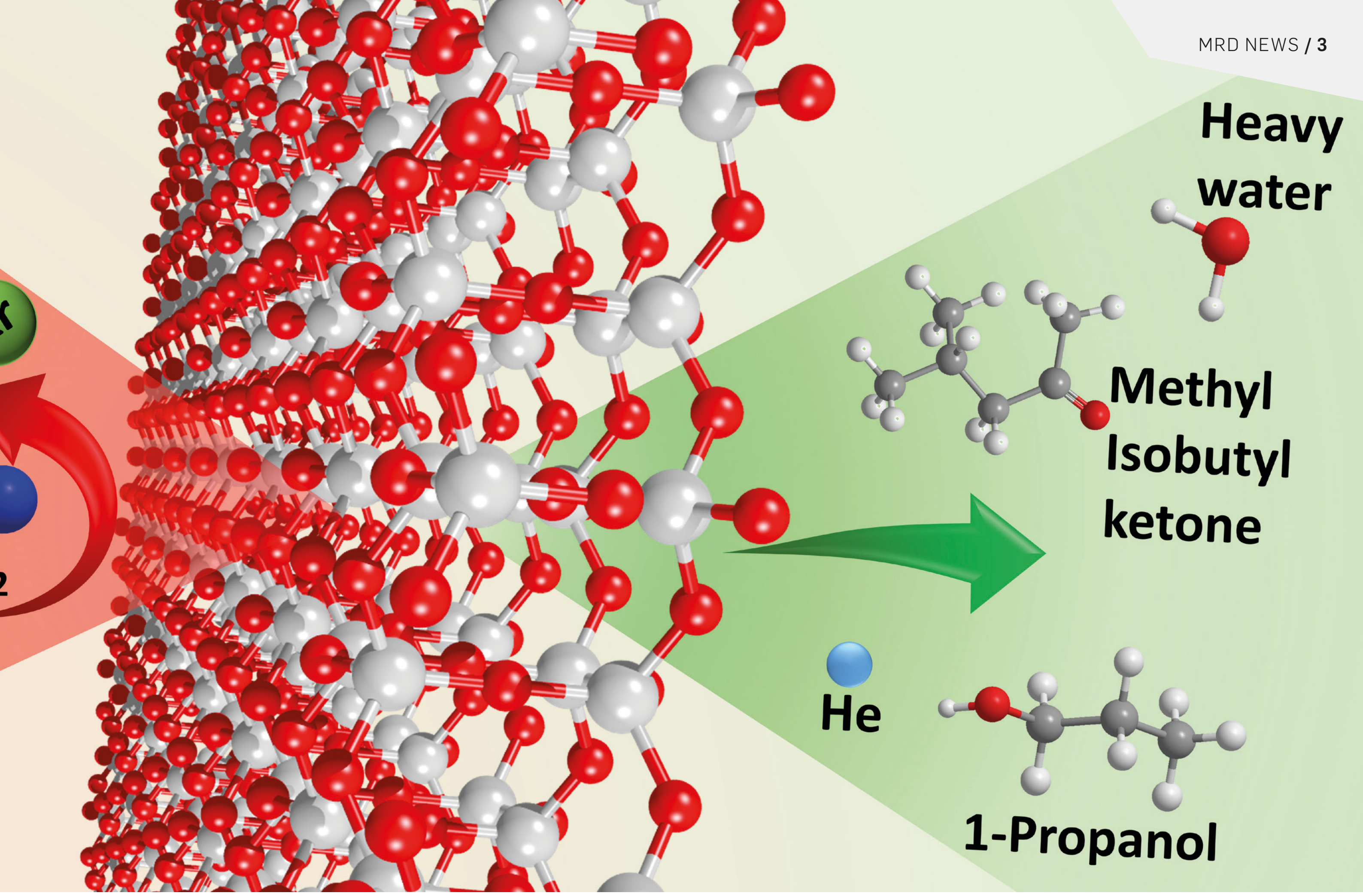


Figure 1: Selective permeation of gaseous molecules with freestanding bilayer silica processed via atomic layer deposition (ALD).

2D-MATERIALS

Molecular Sieving with a Natural Two-Dimensional Membrane

The intriguing properties of the two-dimensional (2D) material graphene and the outstanding success in utilizing it in different technologies ranging from electronics, energy, membranes, biomedical, sensors etc. has aroused tremendous interest in exploring new era of 2D material systems for similar applications. Particularly the transition metal dichalcogenides (TMDCs) and recently extending to other layered and nonlayered material systems has caught the attention of the scientific community especially for sustainable energy storage and conversion. Membranes are very attractive for energy efficient separation and purification systems. They can impede movement of molecules or ions while letting others penetrate by forming a selective barrier. The current interest in membrane technology is to exploit its advantages such as simple operation, low energy consumption compared to



conventional separation routes such as distillation and adsorption. 2D materials with atomic thinness can serve as new building blocks for fabricating ultrathin membranes possessing the ultimate permeation rate. Layers of two-dimensional (2D) silicon dioxide (SiO_2) contains natural pores and can therefore be used like a sieve for molecules and ions. Scientists have been looking for such materials for a long time because they could for example help desalinate seawater and be used in new types of fuel cells.

When a 2D material is pierced with angstrom precision, the resulting nanopores can discriminate chemical species similar to a sieve. There have been many efforts to perforate the 2D material graphene but removing atoms from its crystal structure harms the mechanical stability and it is also hard to control the size of artificial pores. The researchers from Bochum, Bielefeld and Yale have recently studied the permeation of gases in two-dimensional silicon dioxide (2D-silicates) that has intrinsic lattice openings [1]. The 2D material was grown by Atomic Layer Deposition (ALD) on an inert gold surface and was subjected to a detailed spectroscopic and microscopic characterization. After optimizing the preparation conditions, the defect-free 2D membranes were sealed in a vacuum apparatus and probed with respect to the flow of gaseous molecules. It was found that free-standing 2D SiO_2 enabled the passage of vapors like water and alcohols but stopped the atmospheric gases nitrogen and oxygen (Figure 1). The mechanism appeared to be affected much by nanoscale effects.

This study sheds light on the mass transport properties of freestanding 2D SiO_2 upon ALD to grow large-area films on Au/Mica substrates followed by transfer onto Si_3N_4 windows.

These results are exciting because 2D SiO_2 has a high areal density of nanoscopic pores that is simply not possible in artificial 2D membranes. Unlike perforated graphene, the pores in 2D SiO_2 are almost of the same size, and there are so many of them. It acts like a fine-mesh strainer for molecules. Although 2D silicon dioxide is known since 2010, the methodology to synthesize it has been limited in scale and costs. This study is anticipated to widely impact the materials research as such 2D materials with selective permeability are of enormous interest. However, before the 2D silica can be used in practice, it is important to evaluate exactly how many molecules can attach to the surface of the materials or how they can penetrate it, which is part of a newly granted DFG project between RUB and Bielefeld University (Dr. P. Dementyev) in cooperation with Yale University. As membranes are widely used in energy conversion and storage devices, such new 2D porous materials could be at the forefront of aiding sustainable development, for example in the field of energy conversion or storage. The inherent 2D SiO_2 membranes can find further use in seawater desalination and mitigating the environmental problems.

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CIRCULAR ECONOMY OF CRITICAL METALS

A Way for Substitution of Primary Resources

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The development of a circular economy of critical metals is an important requirement for a sustainable future. Advances in electro mobility and the transition to renewable energies increase the demand for critical metals [1]. These metals include rare earth elements such as Neodymium and Terbium which are needed for the construction of highly efficient electric engines and generators. Also, rare and expensive alloying elements like Cobalt, Molybdenum, and Tungsten that are used in high performance alloys can be considered critical. By now, thousand of tons of these materials are dumped while new components are produced from mined primary raw materials. Besides, the depletion of primary raw materials is coupled with high demands of energy and environmental destructions [1]. Furthermore, the locations and the limitation of the reservoirs of the critical raw materials lead to geostrategic dependencies and high economic risks [2].

To overcome this linear type of economy (Figure 1a) and to establish new circular economy systems (Figure 1b), ways to re- or upcycle critical metals are investigated. Today, available recycling strategies are not competitive from an economic point of view [3]. For this reason, there is a need for further investigating and developing recycling methods for critical metals. Our investigations within the research project "GENESIS", funded by the German Ministry of Economics and Climate Protection (Bundesministerium für Wirtschaft und Klimaschutz, BMWK), focus on a powder metallurgical approach to recover critical metals. Scrapped products such as the rare earth element containing components of electric engines or generators can be mechanically crushed to powders that can be used again as secondary raw materials for the production of new components. Other types of scrap are grinding sludge or chips from the subtractive machining of metallic high-performance components which can contain large amounts of critical alloying elements. To recover scrap materials for powder metallurgical processes in particular, efficient methods for sorting, cleaning and crushing to powder material have to be found. The powder metallurgical route for re- and upcycling of the otherwise dumped materials offers a wide range of energy and material efficient manufacturing methods to produce new components from the waste materials. These methods include near net shape sintering and additive manufacturing techniques [4,5].

Field assisted short time sintering processes utilizing impulses of electric current (FAST-SPS) are adapted for the recycling materials and combined with integrated hot forming (FLASH-SPS) to set up desired microstructures and therefore produce high performance neodymium-based magnets at a low energy consumption. Furthermore, the production of high strength steel parts from waste material from subtractive machining by hot isostatic pressing (HIP) is investigated. Thereby, state of the art hot isostatic presses with integrated rapid gas quenching are used to combine the consolidation of the recycling powder with the heat treatments that are required to set up the mechanical properties of high strength steels.

The investigated and further developed powder metallurgical manufacturing techniques can reduce energy intensive production steps like multiple forming, machining and heat treatment procedures in the processing of critical materials. In addition, the goal is to recover the vast amounts of available and already dumped critical metals and keep them in a circular economy to substitute the use of primary resources. This is an important step to a cleaner production of the highly demanded components of a more sustainable future.

“This is an important step to a cleaner production of the highly demanded components of a more sustainable future.”

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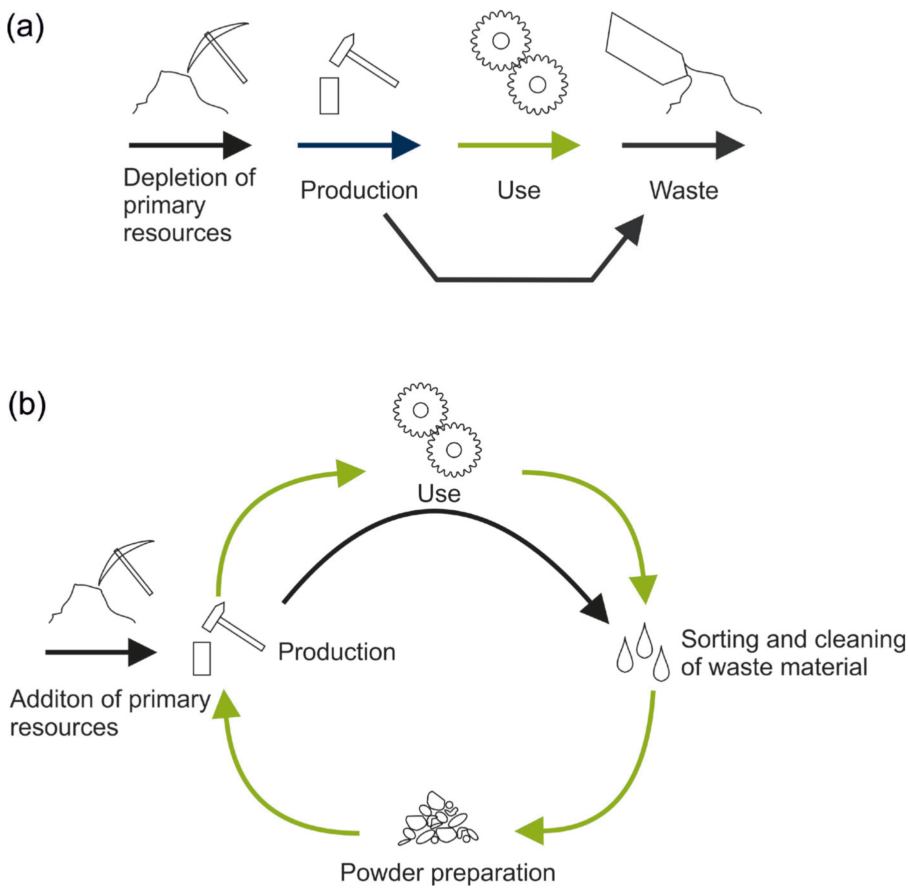
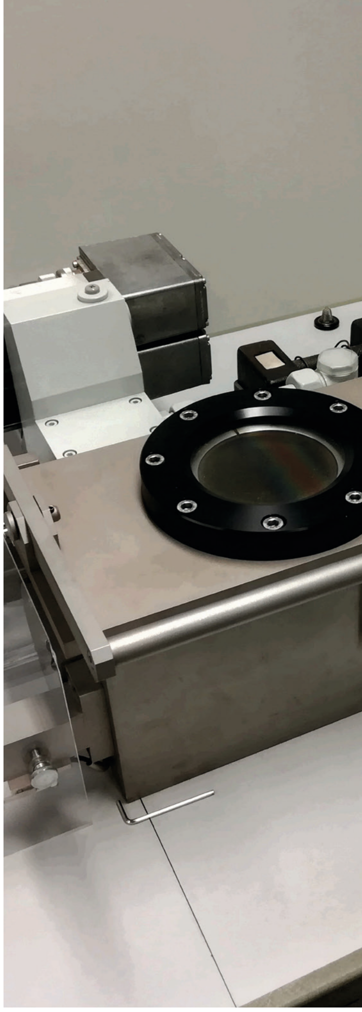
Figure 1: Schematic descriptions of economy systems.
(a) Linear economy.
(b) Circular economy.





ENERGY-EFFICIENT HEAT TREATMENT OF HIGH-ALLOY STEELS

New Simulation Model Considering Chemical Composition and Initial Microstructure

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The metal processing of high-performance tool steels usually consists of a multitude of consecutive manufacturing steps. Besides the forming and machining processes, the heat treatment is one of the most important and energy-intensive steps in the manufacturing process. This is because the properties of the material, and thus those of the manufactured tool, are determined by the microstructure present. The microstructure, in turn, is primarily determined by the chemical composition and the complete processing history. In the case of high-alloy tool steels, the microstructures are typically complex multi-phase systems.

Conventional industrial heat treatment of high-alloy tool steels consists of austenitizing at temperatures above 1000 °C in a natural gas-fired hardening furnace, followed by quenching to room temperature. This is currently followed by a multi-stage tempering at temperatures in the range of 500 °C, typically in an electrically heated furnace. This energy-intensive heat treatment alone can be used to estimate the high energy demand in the production of high-performance tools. The exact temperatures and holding times of the heat treatment of a tool are determined and optimized using a complex experimental test matrix. Subsequently, the heat treatment determined in this way is applied in production for the tool steel and the tool used. However, due to the production process, e.g. recycling of material, there are slight differences in the chemical composition of the starting material, which leads to deviating mechanical properties. In the worst case, this can lead to rejects due to the insufficient mechanical properties of the tools.

Due to the challenges described above, we have developed a heat treatment simulation model using the ledeburitic cold work tool steel X153CrMoV12 (1.2379) as a model alloy. The model considers the chemical composition as well as the microstructure of the starting material (and thus the complete manufacturing route) and calculates the microstructure after heat treatment as a function of the hardening temperature and holding time. The schematic sequence of the simulation model is shown in Figure 1. The model was validated by experimental execution of the heat treatments using a quenching dilatometer (Figure 2). Two different hardening temperatures with six varying holding times each were successfully tested. For a given time-temperature history of the heat treatment, the developed simulation model can compute the carbide dissolution, the concentration of alloying elements in the matrix, and the martensite start temperature. From these data, mechanical properties can be derived.

With the model, we now can simulate local differences between component edge and component core. On the other hand, we can vary the hardening temperature and dwell times to reduce the total heat treatment time while maintaining the same properties and thus increasing efficiency. Furthermore, it is possible to adapt the heat treatment to the changing chemical composition

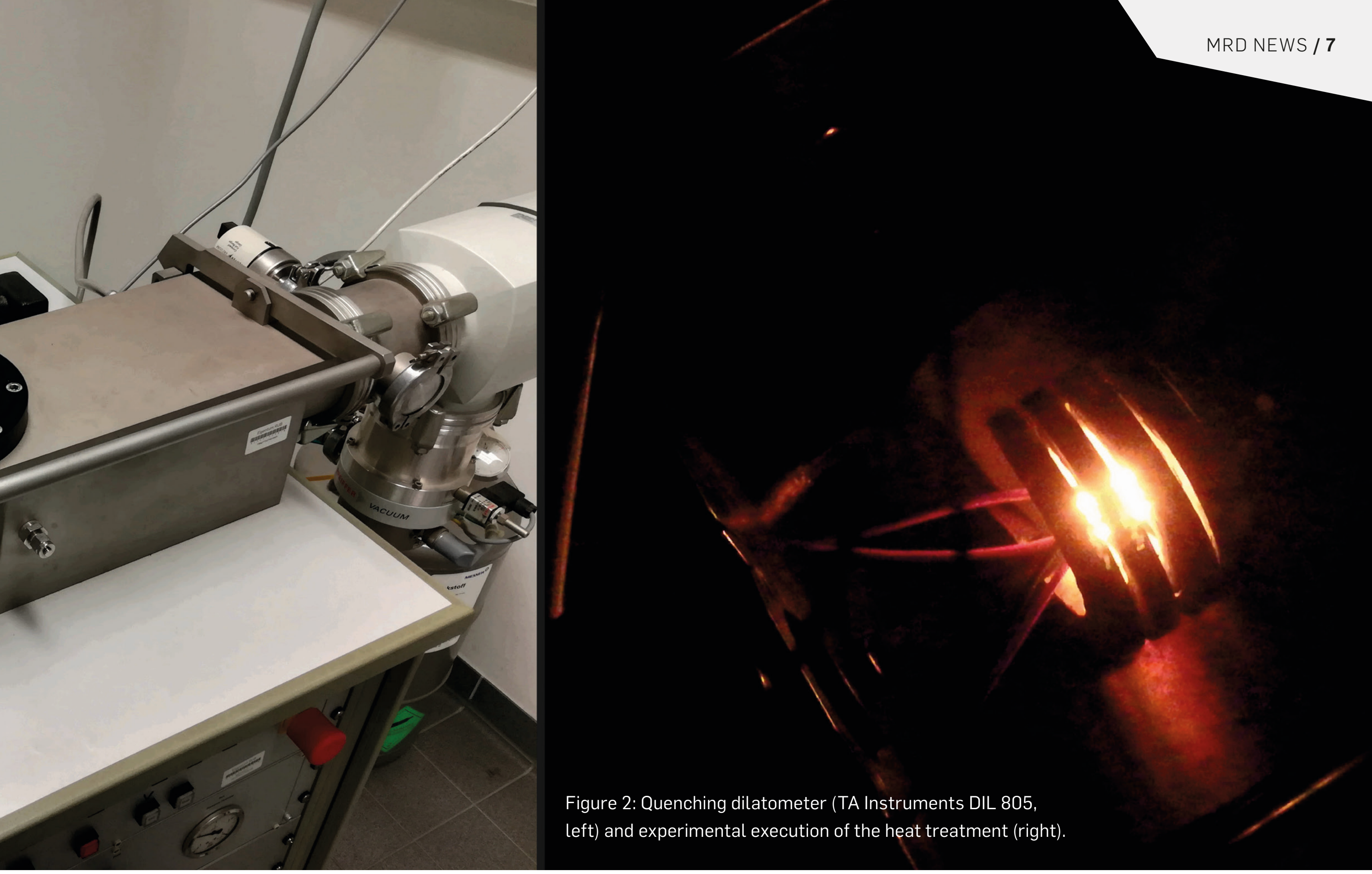
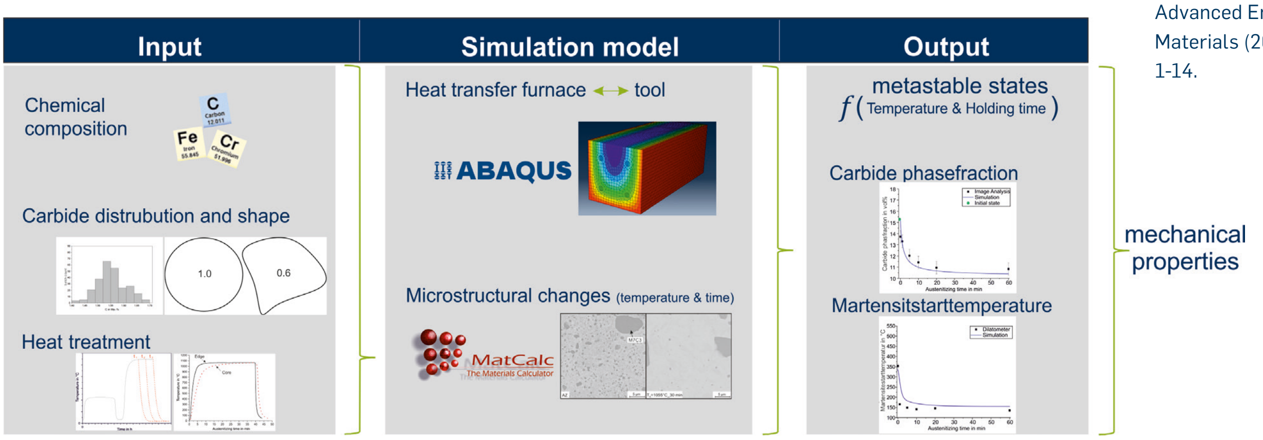


Figure 2: Quenching dilatometer (TA Instruments DIL 805, left) and experimental execution of the heat treatment (right).

of the starting material to guarantee the required mechanical properties and thus the process stability in production. With the help of the simulation model, an alternative heat treatment was developed for a conventional application. The new heat treatment comes along with a lower hardening temperature and requires significantly lower holding times than the conventional process. In addition, this low-temperature heat treatment requires only one stress-relieving tempering step at temperatures of approx. 200 °C, compared to the current three tempering steps at temperatures of approx. 500 °C. In experimental laboratory tests, this developed heat treatment achieves identical mechanical properties, or even surpasses those of conventional heat treatment in other areas. If the heat treatment developed by the simulation and investigated on a laboratory scale can also be used in industrial production, this will result in a significant reduction in the energy required for production. This would lead to a more sustainable production and thus to an optimized overall process chain while maintaining the same product quality.

Figure 1: Flowchart of the simulation model



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METALS RESEARCH ON SUSTAINABILITY AND A CIRCULAR ECONOMY

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Since the dawn of mankind, complex materials have been the backbone of human society. Today, they are indispensable in the fields of energy, industry, transportation, health, construction, safety and manufacturing [1]. With >2 billion tons produced every year, particularly metals stand for massive economic growth, job safety and wealth. Due to the sheer quantities produced and used, they also play a central role in sustainability [2].

Currently, we enter from the age of linear industry into a circular and digitalized economy. This offers huge opportunities to revolutionize the way how production, transport and energy supply work. These changes affect the daily lives of billions of people. Advanced metallic alloys, their production and downstream use, also at large scales, are key to this transition, as they enable a carbon-free, digitalized and electrified industrial and urban future.

Therefore, we devote all our efforts to understand, invent and enable advanced materials and processes for a sustainable and safe future.

More specific, we work on processes, mechanisms and materials for sustainability and a circular economy: The microstructure-centered approach of the department enables us to address and identify pathways towards enhanced sustainability of metallic materials, in areas which include reduced-carbon-dioxide primary production [3,4], recycling of metals, scrap-compatible alloy design, contaminant- and hydrogen tolerance of alloys, hydrogen-plasma based reduction, electrolysis for the reduction of oxides and hydrogen-based direct reduction of iron ores. For this purpose we have designed and modified a number of laboratory-scale reactors in which corresponding experiments can be conducted, under well-controlled reactive boundary conditions and temperature control as well as permanent in-operando monitoring through mass spectrometry.

Topics with high activity are currently hydrogen-based direct reduction of iron oxides, hydrogen plasma-based production plus melting of oxides in electric arc furnaces and the design of alloys that can tolerate highest possible scrap and thus impurity fractions.

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