International Commission on Glass

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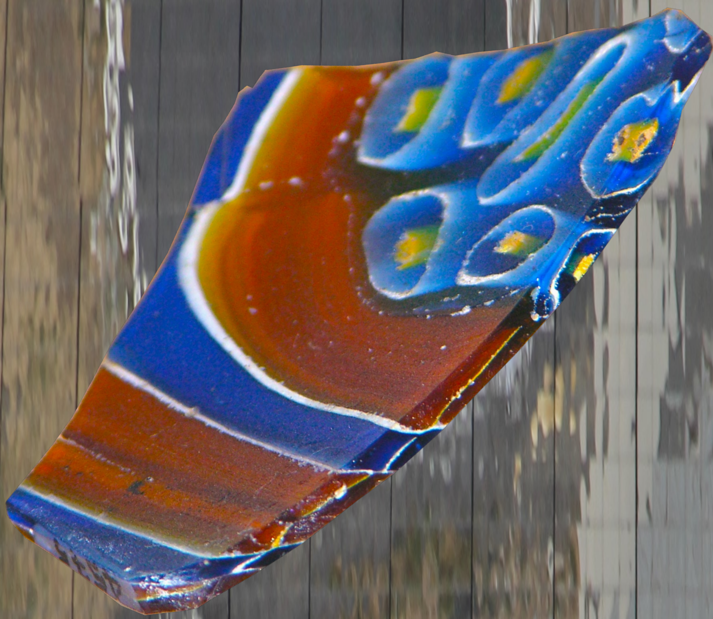
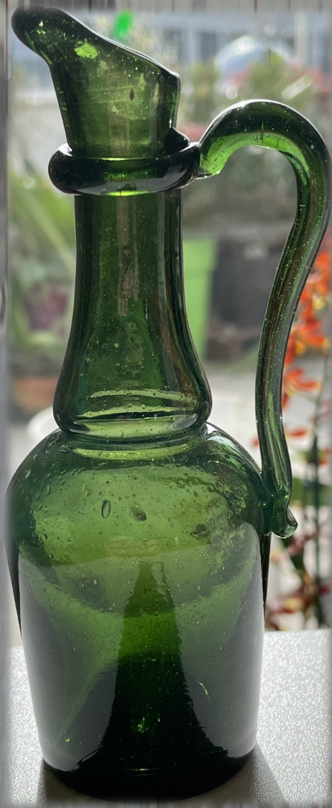
ICG Spring School 2024

Glass for a sustainable future





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What have the Romans ever done for us? Two thousand years of glass recycling

Nadine Schibille * [1]

[1] IRAMAT-CEB – Université d'Orléans, CNRS – France

Urban waste was already a problem in ancient times, and massive landfills are not just a modern phenomenon. Considering the vast quantities of glass used in Roman buildings, however, the amount of glass that has ended up in the archaeological record is surprisingly small. This discrepancy is partly explained by the suitability of glass for recycling, which was extensively practiced since at least the first century CE. Glass recycling gained importance in late antiquity and the early Middle Ages, when the supply of raw glass from the eastern Mediterranean became more difficult and therefore more expensive for political and economic reasons. There is evidence of glass recycling from written sources, archaeological remains and especially compositional characteristics. The chemical analysis of archaeological glass assemblages from the Roman period to the Middle Ages shows evidence of systematic recycling in the form of mixtures of different raw glass compositions, the accidental incorporation of coloured cullet (broken glass) that increases the concentrations of some of the transition metals not otherwise present in the silica source, a loss of volatile elements and/or contamination of the glass batch by the furnace environment, glass-working tools or fuel. This presentation will discuss the existing evidence for glass recycling during the first millennium CE, focusing primarily on the effects of glass recycling on the composition of archaeological glass and its implications.

Keywords: archaeology, LA, ICP, MS, transition metals, cobalt, antimony, contamination

*Speaker

Basic data on glass structure and its influence on viscosity

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[2] IMPMC, Sorbonne University, CNRS, MNHN, IRD, Paris – Sorbonne University – France

After the initial presentation laying the foundations of the glass structure studied since the first diagram proposed by Zachariasen in 1932, using a variety of techniques, we will try to understand how structure and chemical composition influence macroscopic properties, like viscosity variations. In this presentation, we will review the various techniques for measuring viscosities and discuss intrinsic errors, then explain the role of the various elements - network formers, network modifiers and charge compensators - and how they affect viscosity, specific heats and a number of other macroscopic properties.

Keywords: Glass structure, Viscosity

*Speaker
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Glass and glass-ceramic formulation: an introduction

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There is an ever-growing demand for energy to power computers, vehicles, and everything in between. The choices around environmentally responsible energy sources have been hotly debated over the last decade, given concerns about the effects of human activity on our planet. As a response to these concerns, industry is moving towards decarbonization of their energy sources and processes, necessitating transitions to greener fuels, materials, and processes. The glass industry has taken these challenges seriously, vowing to reduce the CO2 footprint and overall environmental impact through process and potential formulation changes. However, current and newly conceived products still need to perform as needed. The glass industry has demonstrated its adaptability before, for example the removal of arsenic oxide from optical glass compositions. By connecting fundamental science through to industrial know-how, it is possible to reimagine glass manufacturing for a sustainable future.
In this presentation, some strategies for formulation of complex glasses and glass-ceramics are offered. Examples are given for nuclear glasses and optical glasses. The focus is on oxide glasses, but complementary examples are also given for chalcogenide and halide systems. A focus is given on effects of different raw materials on properties and processing, as well as on substitutions and tolerance of impurities.

Keywords: formulation, optical glass, nuclear glass, glass, ceramic

*Speaker

From melt to glass fiber

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Glass fibers play a significant role in the green transition in various ways. Enhancing fiber performance necessitates optimizing the process of fiberization from glass melts. To realize this goal, we need to understand: 1) how fibers are formed; 2) how fiber structure changes during formation; and 3) how fiber properties are correlated with fiber forming history. Not every type of melt can be stretched into fibers, making the fiberization process both technologically and scientifically intriguing. In my lecture, I delve into these issues, discussing the dependence of glass fiber properties on both glass structure and fiber forming conditions. I also highlight the challenges encountered in developing high-performance glass fibers. Additionally, I touch upon the quantification of glass fiber spinnability. It should be mentioned that my lecture focuses on oxide glass fibers for both reinforcement and thermal insulation.

Keywords: Melt, Glass, Glass Fiber, Structure, Properties, Fiberization

*Speaker

Towards decarbonized specialty glass

Allison Yake * [1]

[1] Corning Incorporated – United States

The technology brands around the world are now looking for specialty flat glass with optimum performance that also includes sustainable attributes. In addition, glass manufacturers are making validated science-based short- and long-term commitments to reduce their corporate greenhouse gas (GHG) emissions. Using industry recognized methods, estimates and assumptions, we can understand the key contributors of the GHG footprint of these products, which are predominantly energy sources and raw materials. Eliminating embodied carbon requires addressing a number of challenges, including production processes, material composition, transportation, and material sourcing. The input materials, in particular, create further challenges related to composition, material preparation and circularity. All these challenges must be addressed across the value chain with a collaborative mindset and demand transformation in the way we invent, manufacture, and use goods. By providing estimates of the relative magnitude of GHG sources and practical examples, this talk will provide a view of the opportunities and requirements that must be addressed in the journey towards zero-embodied carbon specialty glass.

Keywords: GHG, decarbonize, embodied carbon, specialty glass

*Speaker

Decarbonization Roadmap applied to the Glass Fiber Industry

Anne Berthereau * [1]

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With climate change acceleration, more strict regional legislation and risks for natural resources scarcity, Corporates are reflecting about how to execute their sustainability agenda with more speed, while keeping on balancing for financial, environmental and social performances. For Owens Corning which has been proactive in establishing clear Sustainability goals ahead of many Fortune 500 Corporates, sustainability has recently reached another dimension, being now embedded within Company strategies as a key driver for sustainable profitable growth.

Turning this vision for sustainable profitable growth into execution will require 3 major transformations: on Technology, Management System and Culture.

During that conference, we will display a strategic framework applied by Owens Corning in Europe in this journey to net zero, discuss Technology development required as progress recently made.

Keywords: Glass Fiber, Decarbonization, Hydrogen

*Speaker

Technical glass opportunities for a sustainable future and collaboration with academia

Jeffrey Kohli * [1]

[1] Corning Incorporated – United States

The beginning of the glass "industry" occurred at least 3500 years ago in places like Mesopotamia, Egypt, and Syria. At that time, humans did not consider firing ceramics or melting glass a climate related issue; their struggle would likely have been in generating the heat that was required to fuse raw materials, make a homogeneous glass-forming melt, and form useful or aesthetic articles. Eventually, the creation and industrialization of glass articles such as vessels, windows, light bulb envelopes, and optical communications fiber were life-changing innovations. Today, with widely acknowledged global climate change, we in the glass industry are seeking new technologies that will combat climate change. More sustainable processes and glass products will lead to a new wave of life-changing innovations which are needed to sustain the planet and its inhabitants. Academia and industry have a unique capability, capacity, and responsibility to address this new challenge. The presenter will describe the underpinnings of the challenge in the glass industry and the means for us to work together, to provide life-changing innovations for our world yet again.

Keywords: Sustainability, glass, industry, academia

*Speaker

DECARBONIZED ENERGY ROADMAP – Moving Away From Fossil Fuels

Emre Dumankaya * [1]

[1] SISECAM – Turkey

As the global demand for fosil fuel continues to rise and environmental regulation targets are becoming increasingly stringent, glass manufacturers are faced with the challenge of reducing costs for melting glass under a variety of operating constraints without compromising glass quality and productivity. This enabled a stronger demand among glass manufacturers to invest in innovative energy and emission reduction technologies.
The core glass industry, which has a production process with high energy consumption, produces approximately 95 million tons (Mt) of CO2 per year.Of these emissions, 40-60% are emitted directly from combustion of fosil fuel, 10-20 % come from primary generation of electricity used on site and 25-35% are released from the decomposition of carbonate raw materials. Whilst the sector has made progress by halving emissions in the last 50 years, there is a need to urgently accelerate efforts to increase energy efficiency and reduce CO2 emissions to meet the 2050 carbon commitments.

As many furnaces due to be installed in the coming years will be expected to run for up to 12-15 years, new low carbon fuel technologies need to be proven technically and economically within the next 10 years if the glass sector is to fully decarbonise by 2050. Therefore, to effectively decarbonise the entire sector as fast as possible, it is recommended that the following scenarios need to be investigated and developed by the Glass Sector in order to maximise the chances of successfully decarbonising manufacturing process by 2050:

- Hydrogen
- 100% electric melting
- Biofuels
- Hybrid-fuel scenarios
- Carbon capture

In this speech, alternative solution scenarios and the pathways that can be created will be presented and discussed.

Keywords: decarbonisation, sustainable glass manufacturing, energy roadmap, hydrogen in glass melting, electrification in glass furnaces, carbon capture

*Speaker

Structural design of borosilicate-based nuclear waste glasses

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Borosilicate glasses have a unique blend of processing and product characteristics, making them the most suitable materials for immobilizing nuclear waste worldwide. Historically, nuclear waste glasses have been designed using trial-and-error approaches followed by the development of data-driven empirical models to predict their properties and performance. However, this approach has two significant drawbacks: (1) the conservative nature of the empirical models does not allow the design of glass compositions with enhanced waste loadings over a broad composition space, and (2) being empirical in nature, this approach is unable to explain the deviation in results from a predicted trend. To overcome these challenges, we need to transition beyond the trial-and-error-based approaches and establish a fundamental understanding of the underlying compositional and structural drivers controlling the processing, properties, and performance of the borosilicate-based nuclear waste glasses. In this lecture, the structural design of borosilicate-based nuclear waste glasses will be discussed from the perspective of (1) suppressing the crystallization of aluminosilicate phases, (2) increasing sulfur (as SO42-) and molybdate solubility in the glassy matrix.

Keywords: glass, fundamentals, nuclear waste

*Speaker

How does the redox affect structure and properties? Implication for glass industry

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[2] IMPMC, Sorbonne University, CNRS, MNHN, IRD, Paris – Sorbonne University – France

The first two presentations focused on the structure and properties of glasses and liquids, but what occurs at high temperatures in a furnace or a volcano?
During a period of energy transition, how does the transition from gas to electric or hydrogen furnaces affect the high-temperature properties within a furnace? How do factors such as pressure, temperature and oxygen fugacity influence the conditions of melting and annealing, and how do they affect the properties of the final product?

In this presentation, we will discuss the variability of oxygen fugacity within furnaces, and explores its potential influence on high-temperature properties?

Keywords: Glass structure, REDOX

*Speaker
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The role of chemical diffusion in glass processes and related challenges for glass sustainability

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[1] Surface du Verre et Interfaces (SVI), Unité mixte CNRS/Saint-Gobain – CNRS : UMR125 – France

Chemical diffusion plays a significant role in various facets of the glass melting process, including the assimilation of glass batch remnants into the molten glass and the corrosion of furnace refractories. Predicting the kinetics of chemical diffusion poses several challenges, primarily stemming from the intricate structure of the glass network, which leads to profound interconnections between the movements of different chemical species.
In this presentation, I will explore the characterization and description of chemical diffusion, delving into its theoretical underpinnings and highlighting practical scenarios where diffusion is a crucial factor.

Keywords: chemical diffusion

*Speaker

Modern Computational Methodologies for new glass developments

Alfonso Pedone * [1]

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This lesson will delve into advanced techniques employed in simulating the structure, properties, and behavior of multicomponent oxide glasses. The discussion will encompass both ab initio methods and classical Molecular Dynamics Simulations, providing participants with a comprehensive understanding of the computational tools driving contemporary glass research. The presentation will also highlight recent breakthroughs in machine learning potentials tailored for oxide glasses. Attendees will gain insights into how artificial intelligence techniques enhance predictive modeling, contributing to a more efficient and accelerated exploration of novel glass formulations.

Furthermore, the lesson will cover cutting-edge sampling techniques, particularly focusing on metadynamics. The application of metadynamics has proven instrumental in studying crystallization processes originating from silicate melts, offering valuable insights into the dynamics and thermodynamics of glass transformations.

By the end of the session, participants will be equipped with a nuanced understanding of the synergistic application of ab initio simulations, classical Molecular Dynamics, machine learning potentials, and metadynamics in the realm of glass science. This knowledge is poised to catalyze advancements in sustainable glass development, aligning with the overarching theme of the spring school dedicated to a sustainable future for the glass industry.

Keywords: Molecular Dynamics Simulations

*Speaker

Development of new furnace technologies (H2, electric, blend) and constraints

Malte Sander * [1]

[1] Glass Service – Czech Republic

Today's main challenge is freeing the world of fossil dependency and finding effective solutions for traditional processes. The glass industry uses a high-temperature process to transform raw materials into melt. In this process, the primary energy sources are oil and natural gas. Currently, all-electric and hybrid furnaces with large electric heating systems are slowly replacing traditional regeneratively-fired furnaces. Here, the higher heating efficiency of direct electric heating, compared to the combustion process, reduces the specific energy demand for glass production. However, today's energy mix with an average carbon footprint between 400 and 1000 gCO2e/kWh restrains the decarbonization potential of the glass sector. Therefore, the decarbonization of the energy sector, especially the availability of renewable energy, directly impacts the potential for decarbonization in the glass industry. In the coming years, the choice of melting technology will be heavily influenced by the local availability of renewable energy, green hydrogen, and other decarbonized fuels. This underscores the need for furnace designs that can adapt to local circumstances. In the long run, the glass industry will embrace and develop technologies that allow for high electric shares in the melting process, as electricity is projected to be the most cost-effective energy source. Computational fluid dynamics simulations will play a crucial role in these developments as they help to understand the impact of design changes and can give valuable insight into optimal process control.

Keywords: Decarbonization, electric heating, hydrogen, furnace design, technology

*Speaker

Electric melters: Principle, design and limitations

Ankith Santosh *† [1], Luuk Thielen [1]

[1] CelSian – Netherlands

The glass industry has been undergoing a paradigm shift towards sustainable and energy-efficient practices. One significant advancement in this direction is the adoption of full electric glass melters for large-scale production.
The principle of full-electric glass melters revolves around the utilization of electrical energy as the primary heat source for the melting process. This departure from conventional fuel-based melters not only reduces direct CO2 emissions but also enhances process controllability and efficiency. This presentation brings to light the fundamental mechanisms at play in full electric glass melters, shedding light on the dynamic interactions between electrical heating, heat transfer mechanisms and the fluid flow within the melter.

This understanding of the melting process can help answer questions such as: Will the throughput meet the production demands for high-volume glass types? How adaptable are electric melters to changes in production requirements and variations in raw material inputs for different glass types? What are the challenges and limitations associated with specific glass compositions? These different topics pertaining to energy, quality, melting and costs can best be answered with the help of CFD modeling, specifically using CelSian's GTM-X software that is devoted to the modeling of glass furnaces and processes. Together with an illustration of the capabilities of using GTMX to model electric melters, the presentation also aims to elucidate the dedicated batch model that GTM-X utilizes for the accurate prediction of the batch blanket and its associated impact on the glass melt.

Keywords: Electric melting, batch model, modeling

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Current technologies nuclear waste vitrification furnaces / Technology and issues

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A cold crucible induction melter is in operation in La Hague plant, which is operated by ORANO since 2010 for the vitrification of High-Level-Waste (HLW) arising from decommissioning and dismantling operation and old reprocessing of high Molybdenum content fuel. The well-known advantages of the cold crucible compared to the hot metallic inductive melter are (i) a higher elaboration temperature (ii) an extended lifetime and (iii) a better homogeneity obtained by mechanical stirring and gas bubbling. As a result, the global production capacity is expected to be higher as well as the Platinum Group Metal (PGM) particles concentration in the glass. A part of the development of this technology is made with the help of numerical simulation of the glass flow and heating by direct Joule effect thanks to the high frequency induction power unit. In this paper, the last effort of 3D modelisation of the Platinum-Group-Metals (PGM) particles behavior in the glass and chemical reaction kinetics of the feed are detailed.

Keywords: Cold crucible technology, electric induction heating

*Speaker

Glass synthesis: Focus on Industrial and nuclear glasses

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[2] CEA – CEA, DES, ISEC, DPME, University Montpellier, Marcoule, France – France
[3] UCT Prague, Czech Republic – Czech Republic

Understanding of the reaction steps that occur during glass production is crucial for the optimization of the melting process and to avoid the presence of heterogeneities in the produced glasses (bubbles, undissolved grains, crystals, separated phases). In this presentation, we will describe the physico-chemical mechanisms that lead to the formation of a homogeneous liquid in industrial glass melting furnaces (float glass in particular) and in nuclear waste conditioning processes (French and American cases). We begin by describing the reaction mechanisms involved in the production of French and American nuclear glass. We will then focus on the phenomena implied in the production of a soda-lime-silica glass made from silica and sodium and calcium carbonates. We will show what reaction paths are possible to achieve the desired liquid composition, and why it is crucial to control the properties of the precursors used (particle size, microstructure, melting and decomposition temperatures) to obtain a homogeneous liquid. These explanations will be based both on knowledge of phase diagrams and on in situ analyses at temperature, enabling us to characterize changes in compositions and morphologies (tomography, environmental SEM) and thermal effects (ATD/ATG) associated with melting and decomposition reactions. Finally, we will discuss the contribution of physico-chemical modeling to optimizing the glassmaking process.

Keywords: Glass melting, industrial glass, nuclear glass

*Speaker
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Glass for batteries

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The lithium-ion battery (LIB) has been the dominant technology in the rechargeable energy storage market for more than twenty years. However, to meet the increasing need for electric vehicles and portable electronics as well as stationary electricity and grid-scale energy storage, there is an emerging demand for alternative battery technologies.
The transition to clean energy requires the introduction of energy storage devices with excellent electrochemical properties that respect economic, environmental, and social aspects. Analysis of issues associated with liquid electrolytes led scientists in the past to consider solid-state electrolytes, which made it possible to apply a metal lithium anode and design all-solid-state batteries (ASSB) (Figure 1, (BUBULINCA 2023)).

The typical battery architectures for the conventional lithium-ion and solid-state batteries are represented Figure 2 (JANEK 2016).

Conventional lithium-ion batteries (LIB, Figure 2.b.) contain a porous anode (negative electrode typically made of graphite, grey circles) and a porous cathode (positive electrode typically made of a layered transition metal oxide, violet circles) as 'active' storage components, coated on thin copper (negative electrode) and aluminum (positive electrode) foils, serving as current collectors. A thin separator (grey band, about 10 μm thick) is placed between the much thicker electrodes (each about 100 μm thick). A liquid electrolyte infiltrates the porous electrode and separator assembly, providing fast ion transfer between the electrodes and preventing electronic short-circuiting.

In a lithium ion solid-state battery with a conventional anode (LI-SSB, Figure 2.c.), the liquid electrolyte in the electrodes is completely replaced by solid electrolyte (dark orange circles) in the electrodes and the electrolyte-filled separator is replaced by another or the same solid electrolyte (orange circles). Only with a lithium-metal anode (light yellow) that has a theoretical energy density of 3,700 mA g–1, can a significant gain in energy density be achieved. Changes in energy density are estimated based on the density increase from liquid to solid, considering the high specific capacity of lithium metal (LiM-SSB, Figure 2.a.)) and complete replacement of the graphite and anode electrolyte.

The price of lithium and its resource increasing day by day, there is an emerging need to develop alternative technologies. Sodium ion batteries SIBs are an alternative choice to LIBs. They share similar operating principle as LIBs, they are based on cheaper and abundant raw materials, the electrolytes are water-based and the copper collectors at the anode can be replaced with aluminum collectors. So All-solid-state sodium batteries (ASSSBs) using nonflammable solid-state electrolytes (SEs) and earth-abundant sodium metal are also attracting worldwide research attention. (CHI 2022)

*Speaker

All-solid-state sodium and lithium batteries (respectively ASSSBs and ASSLBs) are promising candidates and could provide high power density with good safety and cycle durability, making them a potential next-generation battery technology. As a result, the identification and optimization of suitable cathode materials and solid electrolytes (SSEs) are essential for developing the practical application of ASSBs.

After years of development, SSEs can be divided into three categories: inorganic solid electrolytes (ISEs), polymer solid electrolytes (PSEs) and composite solid electrolytes (CSEs). Among them, ISEs can be divided into amorphous glass, glass-ceramic and polycrystalline ceramic. Glass is an amorphous super-cooled liquid, whereas glass-ceramics are partially crystallized glasses, consisting of a mixture of a crystalline phase and an amorphous glass phase (SAKAMOTO 2010). The percentage of crystalline phase present can vary across a wide range, typically in the range of 10–90%. The main advantages of glass-ceramic materials are their dense, non-porous microstructure, and good mechanical, electrical and thermal properties. Glass-ceramic SSEs have become one of the hot research directions for SSEs due to their excellent ionic conductivity, electrochemical properties and better compatibility with electrodes. Apart from the obvious advantages of eliminating the liquid electrolyte and associated problems, such as dendrite growth and liquid leakage, the use of solid electrolyte also provides the benefit of removing the need for a separator (e.g., porous polymer membrane), which in turn reduces the cost and fabrication complexity. The use of a solid electrolyte is not without its challenges. At present, the major challenge is the development of materials with high ionic conductivity at room temperature and improved chemical and electrochemical stabilities. (LIN 2023) (VIALLET 2019)

The energy density of batteries will depend upon the capacity of cathode materials. Many researchers developed various cathode materials for SIBs. It is particularly important to develop cathode materials with sufficiently large interstitial spaces within their crystallographic structures to host ions (Li+ or Na+) and achieve satisfactory electrochemical performances of SIBs. Researchers have developed new class glass and glass-ceramic materials that have improved electrochemical performance and cycle life for use in the development of ASSIBs. As a result of the controlled crystallization and evolution of variable proportions of crystalline and glassy phases, glass-ceramics cathodes can significantly outperform conventional crystalline cathodes in terms of superior mechanical properties, good formability, strong electrochemical stability, high ionic and electronic conductivity, and chemical resistance to volumetric changes upon dissolution of alkali metal cations. These properties make them promising candidates for use as cathode materials in next-generation. (GANDI 2022)

The aim of this presentation is to give an overview of glassy and glass-ceramic solid electrolytes and active materials (anode and cathode) for lithium and sodium technologies.

After a description of an ASSB and the requirement, in terms of solid electrolyte and processing, we will present the synthesis and characterization of glass and glass ceramic materials and will provide a review of ionic conductors and describe the current state of research and development of All-solid-state batteries.

The challenges and opportunities associated with these glass and glass-ceramic systems, and All-Solid-State batteries, will be discussed.

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Keywords: glass, glass ceramic, all solid state batteries, Li ion batteries, Na ion batteries, solid electrolytes

Self-healing high-temperature functional glass for hydrogen fuel cell sealing

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Glass and glass-ceramics have shown potential as sealants materials for solid oxide fuel cell (SOFC) and solid oxide electrolysis oxide (SOEC). SOFCs are subjected to different thermal cycles, which required that the sealant materials have long term thermal stability and are able to repair the damages due to long term operation. It is reported these damages can be repaired by the flow of glass in the micro-cracks. However, sufficient flow of glass takes place at elevated temperature, which can deteriorate the other components of the SOFCs. Therefore, it is desired to repair the damages at lower temperature.

The self-healing in materials science is defined as the ability to recover the mechanical integrity and initial properties of a material after destructive actions of external environment or under influence of internal stresses. Self-healing has been claimed to enable an increase of the operating duration of glass seals for SOFC/SOEC. The self-repairing effect is obtained simply by heating the sealing glass above its softening temperature. This effect was shown to operate also in glass-ceramic sealants, provided that the amount of residual glass is enough to enable softening and healing.

In this lecture is presented our work both on non-autonomous and autonomous self-healing processing in glassy materials. It will be illustrating with some examples of self-healing involving glasses in the high-temperature sealing of SOFC/SOEC.

Keywords: Glass sealant, Self, healing, SOCs, High temperature

*Speaker

Challenge and progress in solar mirrors and glass for greenhouses

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With the emergence of the world population, massive urbanization, climate change, and shortage of sustainable natural energy resources, key technologies must be developed to address these vital needs and glass can profoundly contribute to overcoming these challenges. High durability industrial solar mirrors are today used in concentrated solar plants (CSP) to effectively utilize the sun power for heat and electricty generation and the longevity of these plants are well connected to the durability of the solar mirrors. Here AGC is offering one of the most performant and durable solar mirrors which have been already selected by several key CPS projects across the world. On the other hand, looking at the food safety, world population will hit 10 billion people by 2050 which requires 75% more food production by then. However, considering the total arable lands available, global warming, and water scarcity, one can simply conclude that outdorr agriculture would not be sufficient to fulfill this global need. The only viable solution for this purpose in controlled environment agriculture which is today known as greenhouses where you can increase the productivity of vegetable production by 8 times compared to the outdoor cultivation. Nevertheless, reaching to this level of productivity performance neccessitates creation of the correct level of light & climate inside greenhouse which is linked to significant energy consumption. For this matter, AGC has developed the fist economically viable energy saving glass for greenhouses which does not reduce the light transmission, very improtant for crop productivity, while saving annually around 25% of the energy consumed in greenhouse.

Keywords: Solar Mirror, Horticulture Glass, Energy Saving Glass

*Speaker

Observing mechanical and elastic properties of glasses by spectroscopy methods: toward weight-lightening of glass.

Dominique De Ligny * [1]

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Weight-lightening of glass leads both on lower consumption of raw materials and less energy is required for the melting. However, this needs to be compensated by coatings, thermal or chemical reinforcement to increase the mechanical properties and crack residence. In this sustainability approach, it is then key to estimate stress in situ. New development of Raman, Brillouin and optical spectroscopies allow evaluating directly residual stress and mechanical modified glass structures.

Keywords: residual stress, densification, Raman, Brillouin, luminescence of Rare Earth Elements

*Speaker

responsible and secure supply of raw materials

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We need more and more metals. At the start of the 19th century, our societies used only a few metals (iron, copper, zinc, lead, etc.), but today almost all of the elements of the Mendeleïev table is found in various physico-chemical forms in our daily lives (nickel sulphates, rare earth oxides, aluminium, etc.).
All the technologies at the center of our economies are metal-intensive. Such is the case for energy transition technologies (solar, photovoltaic), electric mobility technologies (batteries, hydrogen fuel cells) and digital technologies (optical fibres, electronic devices). Industrial glass, which is used in some of these technologies, is also affected by these metal consumption needs: Si, B, Li, Ti, etc.

The growing demand for metals is coming up against an increasingly limited supply, particularly for European consumers. European countries have limited mining production or processing plants on their territory, and are therefore dependent on other countries for their supplies. Global geopolitics are tending to crystallise now, making supplies less and less secure.

At the same time, the mining sector is facing increasing opposition because of the significant environmental and social impacts still being generated at many production sites around the world. The challenge for the sector is dual: to meet rising demand while producing responsibly. To produce in a responsible way, it will be necessary to limit greenhouse gas emissions, control water consumption and integrate site restoration into the project design phases.
This talk will address all these aspects, to ensure that the industrial glass sector can be supplied safely and responsibly.

Keywords: raw materials, supply, responsible

*Speaker

The contribution of thermodynamics in determining the parameters of elaboration and energy efficiency

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A glass melter fulfills two main functions: it enables heat exchanges between the energy source (combustion and electric boosting), the raw materials, and the melt. It is the vessel for the chemical reactions transforming the raw materials into a melt. Following the principle of energy conservation, the energy balance of a furnace can be established. Its performance can then be evaluated by benchmarking it against other furnaces.
Once the relative position of a furnace is established, a new set of questions arise: how could the energy efficiency of the furnace be improved? Could the pull rate of the furnace be increased? Are these improvements possible while maintaining sufficient glass quality, and without shortening the furnace lifetime?

A large part of these questions can be answered by using a 1-dimensional thermodynamic approach well described in Pr R. Conradt's papers. The efficiency of this elegant approach will be demonstrated to show, for example, the influence of changing the raw material selection, cullet fraction, electrical boosting, and pull rate on the energy efficiency of a glass melter.
This approach is however limited when it comes to ensuring glass quality, and especially bubble removal. In production, bubble upsets are often related to a wrong temperature and glass flow management, which allows poor-quality glass melt to exit the furnace. Industrial examples of how such issues are observed and solved using Computational Flow Dynamics will be shown.

Keywords: energy efficiency, thermodynamics, glass melting

*Speaker

Refining techniques and developments

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The glass-forming liquid is melted industrially in large open tanks. The raw materials are added in granular form. Melting and chemical reactions between the grains produce a silicate liquid with a large amount of impurities, mainly bubbles. Since the rising velocity of a bubble is proportional to the square of the bubble radius and inversely proportional to the dynamic viscosity, the residence time of bubbles in the glass bath can be long. The classical method of bubble removal is to increase the melt temperature up to 1450C. This reduces the dynamic viscosity. The introduction of fining agents in the raw materials leads to the release of dissolved gases in the liquid, which inflate bubbles by mass transfer, especially if the temperature is high enough.

In such a process the mass transfer is crucial to analyse. The beginning of the lecture will be devoted to recalling the main characteristics of the mass transfer between the bubble and the glass-forming liquid. A list of the main gas species involved in the mass transfer will be given. It will be shown that while the fluid mechanics is in the creeping flow regime, the mass transfer is driven by advection. Some data on equilibrium constants of oxidation-reduction reactions and gas solubilities will be recalled. A description of oxidation-reduction reactions of fining agents is also given. Mass transfer between liquid and bubble will be presented with emphasis on the determination of Sherwood numbers, the dimensionless number of mass transfer coefficients. Emphasis will be placed on the chemical coupling between the liquid and the dispersed bubble phase.
A numerical model will be presented to study the mass transfer and bubble release, taking into account the coupling with oxidation-reduction. Some numerical examples will be used to show the main differences between the thermodynamic equilibrium and non-equilibrium states.

The innovative refining techniques will be presented in the last part of the talk. The removal of bubbles is controlled by the bubble size, the driving force is due to the concentration difference of the gas in the bulk and at the surface. Reducing the pressure increases the concentration difference, leading to an increase in the rate of bubble removal. Few numerical tests will show the effect of pressure reduction on bubble removal. The other possibility is to introduce a permeable gas species such as helium. The numerical model will be used to study the effect of helium. Finally, centrifugal fining will be presented in the last part of the talk.

Keywords: glass melting, bubble removal, oxidation, reduction reactions, mass transfer

*Speaker

Life Cycle Assessment approaches developed for glass sustainability

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In recent years, the importance of environmental sustainability has become increasingly evident across various industries. This growing awareness has prompted businesses to reevaluate their operations and embrace more eco-friendly practices. As a result, Life Cycle Assessment (LCA) have gained significant traction being a systematic and comprehensive methodology used to assess the environmental loads, caused by use of resources, energy and environmental consequences of pollutants released into the environmental compartments associated with all stages of the life cycle of products, processes and services from raw material extraction through to disposal. The International Organization for Standardization (ISO), provides guidelines for conducting a Life Cycle Assessment within the series ISO 14040 and 14044.
ISO states that LCA methodology is able to i) identify opportunities to improve the environmental performance of products at various points in their life cycle, ii) inform decision-makers in industry, government or non-government organizations (e.g. for the purpose of strategic planning, priority setting, product or process design or redesign), iii) the selection of relevant indicators of environmental performance, including measurement techniques, and iv) marketing (e.g. implementing an ecolabelling scheme, making an environmental claim, or producing an environmental product declaration).

Therefore, this analytical tool provides industries with valuable insights into their environmental performance, enabling informed decision-making towards more sustainable practices. In the industrial sector, LCA holds immense potential as a strategic approach to assess and optimize the environmental footprint of products and processes. By quantifying environmental impacts across the entire life cycle, industries can identify opportunities for efficiency improvements, waste reduction, and the adoption of cleaner technologies. In addition, it is possibile to facilitate the comparison of alternative production methods, materials, and design choices, enabling companies to make informed decisions that minimize environmental burdens while maximizing economic and social benefits.

Unlike the predominant environmental certifications currently proliferating within industrial contexts, such as those focused on Carbon Footprint or Environmental Product Declarations (EPD), the Life Cycle Assessment (LCA) methodology offers a more holistic approach encompassing a wide array of environmental indicators. This inclusivity enables a unique and holistic perspective on environmental sustainability.

Among the industrial sectors, the glass industry plays a significant role due to its widespread use in packaging, construction, and consumer goods. Understanding the environmental impacts associated with glass production and recycling is crucial for implementing sustainable practices.

After a preliminary description of the fundamentals of this methodological tool with reference to

*Speaker

some aspects still under discussion in the international scientific community, some case studies relating to the functionalisation of self-cleaning float glass and the recycling of glass waste in the manufacturing of ceramic glazes will be examined.

The construction sector significantly influences energy consumption, material usage, and consequent environmental impacts. Addressing concerns such as reducing energy consumption and regulating the exploitation of non-renewable resources are crucial aspects to tackle within this sector. Implementing design systems that consider both energy and material usage throughout a building's lifespan, along with optimizing construction systems to maximize energy efficiency and minimize environmental impacts, becomes essential for eco-design. Consequently, the industrial sector advanced the development of engineered nanoparticles (ENPs) for use in an increasingly diverse array of consumer and industrial goods. Leveraging the distinctive physical and chemical attributes of nanoparticles enables the creation of innovative applications. These nanoparticles exhibit unique properties (chemical, mechanical, optical, magnetic, etc.) distinct from their bulk counterparts. Consequently, ENPs have found widespread application across various industrial domains, including the functionalization of different types of surfaces, such as metals, glass, composing architectural and decorative indoor and outdoor elements, to obtain specific surface properties. In particular, nanoparticles such as titanium dioxide, are often incorporated or coated onto building materials to confer additional and enhanced properties such as self-cleaning, antibacterial features, anti-fogging, lightweight characteristics, mechanical robustness, durability, and fire resistance. In the case study, the ecodesign of an industrial scale-up of nanoTiO2 self-cleaning coated float glass production performed by LCA methodology will be discussed, focusing on the assessment of both human health effects and environmental loads of the entire life cycle of this nanomaterial. When exposed to radiation of adequate wavelength, nanotitanium dioxide (TiO2) shows peculiar characteristics like photocatalysis of redox reactions, but also superhydrophilicity and antibacterical properties. The functionalized surface has an improved cleanability and antibacterial activity but it also becomes active, i.e., to reduce the concentrations of air pollutants such as nitrogen oxides (NOx) and volatile organic compounds (VOCs) deposited on or in contact with the material surface. Among several coating methods to create a thin film on glass surface, such as vaccum arc deposition, alkaline hydrothermal method, hydrothermal method and others, in this work, a modified coating method consisting in first a decrease of the initial substrate roughness and then dip-coating of the softened glass into a TiO2 nanosuspension has been used with the aim to produce films with enhanced adhesion to the substrate. The results of the environmental assessment has been discussed in terms of impact and damage categories and an LCA comparative analysis between nanotitania functionalized float glass and uncoated float glass has been analyzed in order to highlight the main critical hotspots.

To explore the recycling-related issues, the use of glass waste in the manufacturing of ceramic glazes will be illustrated. The ceramic industry stands as a pivotal manufacturing areas in the world. With the growth in the economy and the development of the ceramic industry, the need for ceramic products is increasing. These products find extensive application in different sectors, including ceramic tableware, floor and wall tiles, vitrified products, and sanitary ware. Ceramic tiles, in particular, holds a prominent place in the building sector.

With increasing demand for raw materials, production capacities are rapidly increasing, even if accompanied by difficult environmental challenges related to the finite nature of natural capital. It is therefore becoming imperative to curb raw material consumption. Considering the importance of the glassy part in the ceramic glaze, the usage rates of frit-based materials are quite high. Ceramic glaze protects products from external influences and gives the product an aesthetic feature. One of the most frequently used raw materials in the preparation of ceramic glazes is frits that are the main component of the batches used in the composition of ceramic

glazes. They can usually be sold in pure form to ceramic manufacturers who create their own glazes, or in some cases, frit manufacturers produce and supply the glazes themselves.
The environmental effect of the substitution of glass cullet instead of frit used in glaze compositions in the ceramic industry will be discussed and the comparative LCA analysis with traditional formulations will be considered.

Keywords: Life Cycle Assessment, environmental impacts, nanotitania, floeat glass, frits, ceramics

Sustainable raw materials for glass production

Ates Gosterislioglu * [1]

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Glass melting is an energy intensive process with a reported average energy consumption between 4 to 7 GJ/ton, which requires reaching high temperatures to obtain a glass melt by fusing the inorganic oxide raw materials namely; sand, feldspar, limestone, dolomite and soda ash. Combustion of fossil fuels and decomposition reactions of the raw materials that are taking place during melting accounts for the main greenhouse gas emissions, especially CO2. Glass melting process generates 0.6-0.8 kg of CO2 per kilogram of glass produced, ranking it one of the major CO2 emitting processes in globe. Approximately 2/3 of the emissions of the glass melting process originates from combustion of fossil fuels and 1/3 of the emissions arise from reactive and thermal calcination (decomposition) of carbonated raw materials which are limestone, dolomite, and soda ash. Total contribution of CO2 gas originating from decomposition reactions may reach up to 0.2 kg for each 1 kg of produced glass. To prevent irreversible damages and catastrophic effects of global warming, net emissions of CO2 need to decrease about %45 by 2030 and reaching net zero around 2050. This lecture aims to discuss the current situation and future prospects of raw materials used in glass production both from literature and industrial & laboratory experiences.

Keywords: melting, raw materials, sustainability

*Speaker

Sustainability in Glass Manufacturing: Contribution from Silica and Silicates

Hans Van Limpt * [1], Lesley Beyers *

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The European Union is dedicating billions of euro's to the aim of becoming climate neutral by 2050. More than a 100 M€ is allocated to projects in the glass industry and targets carbon neutrality though hydrogen combustion, electric and hybrid furnaces, biofuel and syngas combustion, or carbon capture technologies.1 All these 'net-zero technology' projects focus on alternatives to carbon-based fuels, but not on intrinsic improvements to the melting process itself.

In this paper the relation between the batch melting process and the energy requirement of industrial glass furnaces will be discussed based on the energy breakdown of a float furnace. It emphasises the gains that could be achieved by improving the batch melting process and reduce the average residence time of glass at high temperatures.

To obtain the intended glass quality, an energy-intensive homogenization step is required because the primary glass phases that are formed are of inferior quality. The choice of raw material has a direct influence on the quality of the primary melting phases and the time required for homogenization of the glass, and thus on the energy consumption of the glass furnace. By using alternative silica materials and silicates, the quality of the primary melt phase can be greatly improved and less energy will be required for the homogenization process.

Carneiro (2023) Carbon Neutrality: Funding Projects and Future Challenges. GlassTrend-NGF Seminar.

Keywords: Batch, raw materials, minerals, melting, sustainability

*Speaker

Schott Climate Neutral 2030 – a way to decarbonization in the glass industry

Dr Wolfgang Schmidbauer * [1]

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The transformation from a fossil energy-based glass production to the use of Zero Carbon (ZC) energy within glass production and, furthermore, the subsequent technological roll-out into production is one of the most challenging tasks of today's R&D. SCHOTT, as a global specialty glass manufacturer and leading company for ZC transformation, is pushing the usage of green electricity in glass melting to its limits, for example, within the funded flagship projects PROSPECT F&E and PROSPECT Pilot. These projects will reduce CO2 emissions of a melting tank up to 80% by the use of electric melting. Other alternative ZC energy sources like Biofuel or Hydrogen may be introduced later for a complete carbon-neutral production. Besides that, further innovative ZC technologies like plasma flame or microwave heating show the potential to enlarge the portfolio for technological transformation even further. This talk will deal with some technical insights of existing and potential technological solutions to overcome hurdles in the transformation path of SCHOTT's glass production towards climate neutrality.

Keywords: transformation, zero carbon, glass production, electrical boosting, biofuel, hydrogene, plasma, technological roadmap, special glass

*Speaker

Various ideas and technical foundations for achieving carbon neutral glass melting

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With extreme weather occurring on a global scale and large-scale natural disasters increasing, responding to climate change issues, especially reducing greenhouse gas emissions, has become a common issue worldwide. More than 150 countries and regions have announced that they will achieve carbon neutrality by a certain year, such as 2050.
Flat glass is one of the essential materials for humanity. Architectural glass for creating a bright living environment that protects from the rain and wind and lets in natural light. Automotive glass that ensures a clear field of vision even when moving at high speeds. Display glass forms and protects devices that display a variety of information.

In 2018, sheet glass production in Japan was 160 million m2 (1.2 million tons) per year, and CO2 emissions from the sheet glass industry were 1.1 million tons per year. This is equivalent to 0.9% of Japan's total GHG emissions and 4% of industrial CO2 emissions. This picture appears to be similar on a global scale.

The CO2 emissions for each ton of glass produced in a classic regenerative air combustion furnace is approximately 0.5 ton. This includes 0.34 tons from fossil fuel combustion and 0.16 tons from carbonate raw materials. The result of decades of development and operation by our predecessors, these numbers are not to be faulted. However, we have to let go of the sophisticated combustion-based glass production furnaces we inherited from our predecessors. Achieving both carbon neutrality and a sustainable supply of glass products will require a major global challenge over the coming decades. In this lecture, we will introduce this difficulty by giving some concrete examples.

Table 1 summarizes the means to reduce CO2 emitted from glass production. As mentioned above, the CO2 emissions per ton of glass produced in a classic regenerative air combustion furnace are approximately 0.5 tons, of which 0.34 tons come from the combustion of fossil fuels and 0.16 tons from the carbonate raw material.

Methods for reducing CO2 from burning fossil fuels can be categorized into energy saving (reducing the amount of fossil fuel used) and switching from fossil fuels to low-carbon energy sources. Energy saving measures include the introduction of oxy-fuel combustion, the introduction of various types of waste heat recovery equipment, and the reduction of heat loss from the furnace walls by strengthening insulation. These are already viable technologies, but they alone will not produce sufficient CO2 reduction effects. To achieve dramatic CO2 reduction effects, we must switch from fossil fuels to low-carbon energy sources. Alternative combustion gases are hydrogen, ammonia, and biogas. Electricity can also be used as a heat source other than combustion. Whether it's electricity, hydrogen, or ammonia, it's important to identify how clean it is. We need a transformation strategy that takes into account the CO2 footprint of available

*Speaker

alternative energies by region and by decade.

Raw material-derived CO2 emissions are the result of the thermal decomposition of carbonates such as soda ash, limestone, and dolomite. Soda ash can be derived from both chemical and natural carbonate minerals. Limestone and dolomite are natural carbonate minerals. In the glass melting furnace, CO2 that was originally fixed underground is released into the atmosphere. The highest priority in reducing these emissions is to reduce the amount of raw materials used, i.e. to increase the proportion of recycled cullet. Using cullet collected from construction debris to melt high-quality glass such as float glass is a big challenge, but it has to be done. Glass production using 100% recycled cullet is difficult in terms of market balance, so it is inevitable that mineral raw materials will continue to be consumed. It is possible to use quicklime or calcined dolomite instead of limestone or dolomite. Limestone and dolomite are fired separately in a kiln to transform them into oxides before being put into a glass furnace. Although carbonate minerals emit CO2 into the atmosphere, it should be easier to capture the CO2 emitted by firing them in a kiln alone rather than decomposing them in a glass furnace mixed with combustion gas. Industrially available sources of sodium other than sodium carbonate are sodium chloride or caustic soda. A small amount of sodium chloride may be added to the batch as a fining agent. The use of sodium chloride as the main raw material for soda-lime glass is impractical when considering what to do with the large amounts of chlorine (and probably hydrogen chloride) generated in the furnace. In this lecture, we will examine how to use caustic soda as a raw material for glass and the remaining issues.

The two directions mentioned so far aimed to reduce or eliminate CO2 sources. In addition, it is also necessary to consider capturing CO2 from furnace flue gas, then storing it underground or utilizing it as a valuable resource. The site must either have infrastructure in place to store the captured CO2 underground, or a stable channel to sell the CO2 or its derivatives over the life of the furnace, which exceeds 15 years. In this lecture, as an attempt to clear this difficult condition, we will also consider the idea of local production for local consumption.
Finally, I will introduce some important points from the perspective of industrial R&D involved in the innovation of glass melting processes. We work both to bring new glass products to market and to maintain the sustainable supply of existing glass products. Invention of technology that gives competitiveness to small-volume production of specialty glass. Innovative technology that makes the mass production process of commoditized glass carbon neutral. Both development topics are important to us. Although these may seem to be at opposite ends of the spectrum, the scientific understanding required is common to both types of development.

Keywords: glass melting, glass batch, furnace design, carbon neutral

The usefulness of modelling for improving energy efficiency

Miroslav Polak *† [1]

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The CFD modelling in the industrial processes (glass production as well) is nowadays more than usual.
It is well known that the modelling could be used for getting better insight of what is happening inside the furnace, where normally very difficult inspection is possible due to the high temperatures.

Furthermore, the modelling could be very useful when planning furnace design changes as the simulations give the customers the possibilities to directly see the effects with no harm to the furnace or even the running production. Result of the simulation could even help the customers to find the sources of defects.

In these days there is one word, that is repeated again and again, and the word is decarbonation. There are multiple ways how to achieve this goal. And as always, there is still a huge pressure on a drop of pricing. One topic connects both these tasks – energy efficiency. The higher the efficiency, the lower emissions and the lower the costs for melting the glass.

Again, there are multiple ways to describe and evaluate the efficiency of any process. The simplest way is to produce the same amount of glass (or higher) of the same quality (or better) with lower energy input.

It is obvious that using "trial and error" approach is not a good way as it quickly becomes extremely expensive. And this is exactly the task for mathematical modelling. Many possibilities could be evaluated in short time and with reasonable costs.

Very likely, when any glass producer plans the furnace optimization, the CFD model is used prior the realization.

Furthermore, new concepts are currently being developed. This would be impossible without use of CFD modelling.

Glass Service (GS) has internally developed a software (Glass Furnace Model - GFM) that is aimed at the usage in glass melting industry. It is regularly updated to give the customers as many benefits from modelling as possible. Many customers use GMF on daily basis, other order just the modelling studies directly from GS. The positive feedback from the customers is the leading motivation for us to go on.
There will be shown some examples of using GFM to achieve customer's needs (such as lower carbon footprint, lower energy demand, etc.) in the presentation.

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Keywords: mathematical modeling, CFD, furnace optimization

Improving container glass collection & recycling: a European Perspective

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Glass is one of the only packaging materials that can be infinitely recyclable in a closed bottle-to-bottle loop. Once produced, a glass bottle becomes the primary resource needed to produce new bottles – saving resources, energy and lowering carbon emissions.
For decades, glass has been successfully collected for recycling via kerbside and bottle bank collection across the EU under Extended Producer Responsibility (EPR) schemes. Today, more than 8 in 10 glass bottles in the EU are collected for recycling, and projects such as the Close the Glass Loop partnership aim to increase this rate to 90%.

This session will look into the importance of closed loop glass recycling for the container glass indsutry in terms of decarbonisation and circularity, the main drivers explaining high levels of collection & closed-loop recycling in Europe, what are the remaining challenges and how the glass packaging value chain collaborates to address them.

Keywords: Sustainability, glass packaging, collection, recycling, decarbonisation, partnerships

*Speaker

Posters

https://icg-school2024.sciencesconf.org/

Advancing circularity of composites through recovered glass fiber waste specifications

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One of the composite industry challenges is to find sustainable and cost-effective solutions to use recycled material from end of life composite. Recycling glass fibers as a source of material for remelting is an effective route. It allows thanks to the infinite glass remelting ability, to recover virgin fiber performance and scale for recycling is attractive.
Manufacturing of glass fiber for composite reinforcement requires very high quality of the input material. This includes known and consistent chemical composition, low level of contaminants, either metallic, ceramic or even organic. One of the current challenges to overcome is to define waste class specifications that could be consistently collected.

Thus, bringing back fibers from old composite into glass melters needs to remove barriers in the way we control the quality from the initial end of life material or waste collection to its treatment through various processes to recover the glass fibers.

At Owens Corning, we are committed to building a sustainable future through material innovation, with 2030 goals of reducing by half our scope 1 & 2 emissions of GHG and send zero waste to Landfill.
This poster is talking about recent progress made at Owens Corning when it relates to glass waste collection and specifications for remelting glass fiber from wastes. We will talk about challenges that we had to overcome, the specifications to be developed, the online test measurements and how we have proven that remelting of glass fiber waste is a viable opportunity for true circularity for composites materials.

Keywords: end of life composite, Recycling glass fibers, glass remelting, glass waste specifications, glass fiber manufacturing

*Speaker

Alkali-silica reaction in cement mortars containing glass particles

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The work deals with the possibility of using glass in concrete and summarizes its possible negative effects from the point of view of corrosion in concrete. The critical mechanism that occurs on the interface between the glass grain and the cement matrix is the formation of an expansive alkali-silica gel. Alkali-silica gel is hygroscopic and by further absorption of water into its structure, it creates tension strength in the hardened material, which can lead to its failure. The aim of this work was to investigate the effect of glass shards on the alkali-silica reaction (ASR) in mortars. ASR was measured and evaluated according to the accelerated dilatometric test ASTM 1260. It was found that the fine fraction of glass (63-125μm) has positive effect on ASR mitigation, while the coarse fraction of glass (2-4mm) promotes the formation of ASR due to the easy dissolution of glass in a low pH environment. This statement was subsequently supported by electron microscope (SEM) images, where cracks through the glass grains and the binder phase were observed on samples with a coarse glass fraction. Furthermore, it was found, the higher amount of coarse glass fraction is presented in the sample, the higher formation of ASR was observed.

Keywords: Waste glass, Mortar, Alkali silica reaction, Cement

*Speaker

Can proper selection of alumina source increase the retention of technetium during vitrification of nuclear waste?

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The volatilization of technetium-99 during the vitrification of low-activity waste (LAW) into glass poses a significant challenge. Recycle loops are commonly employed to address this concern; however, they may lead to issues such as sulfate phase formation. Achieving high single-pass retention of Tc (or its non-radioactive surrogate, Re) is thus essential for enabling formulations with high waste-loading. In our prior investigation, we observed markedly enhanced Re retention in a LAW melter feed containing gibbsite as an alumina source compared to a compositionally similar feed using kyanite. To investigate this phenomenon, we formulated representative LAW feeds using different Al-sources-kyanite, gibbsite, boehmite, and corundum-and evaluated Re retention in the resulting glasses. Our findings reveal that boehmite feed exhibits the highest Re retention, closely followed by gibbsite, whereas kyanite and corundum feeds exhibit significantly lower retention. This discrepancy is attributed to the formation of nano-crystalline γ-alumina during heating of feeds containing gibbsite and boehmite. As proposed by Xu et al. (J. Nucl. Mater., 483 (2017) 102-106), γ-alumina might adsorb sulfate-perrhenate melt, which persists in the reacting melter feed following the decomposition of the nitrate-nitrite-carbonate melt.

Keywords: vitrification, Tc/Re retention, alumina

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Cathode properties of Iron Based Oxide Glasses for Sustainable and High-Energy Density Lithium-ion Batteries

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As energy concerns grow and the society needs more sustainable and renewable energy sources, the ability to effectively store and retrieve energy is paramount. Currently, rechargeable lithium-ion batteries (LIB) seem to be one of the best alternatives to reduce our dependency on fossil fuels. LIB are made of cathodes materials based on polycrystalline oxides or polyanion compounds (1). However, some of them have their performance limited by their crystalline structure where other compounds suffer from irreversible phase changes during cycling (2). To overcome these key shortcomings, implement glasses as cathode materials seems to be an interesting approach. Indeed, depending on the glass composition large specific capacities can be reached (3). Moreover, glasses have a structure composed of free volume that can easily accommodate structural changes upon lithium ions extraction and insertion (4). Finally, glass production is scalable and commercially easier to implement than most of synthesis processes of conventional cathodes materials.

In this study, several iron based oxide glasses have been investigated as promising cathode materials for sustainable and high energy density LIB. The influence of the nature of the polyanion (PO4, SiO4) and the synthesis conditions on the structural, electrical and electrochemical properties of the glasses were examined. Electronic and ionic conductivities were measured by Electrochemical Impedance Spectroscopy (EIS) on the bulk glasses. The electrochemical properties in terms of specific capacity, redox potentials vs Li+/Li, first cycle capacity loss, coulombic and energy efficiencies of these materials were investigated in coin-cell (vs lithium metal) by Galvanostatic Cycling (GC). Finally, to elucidate the reaction mechanisms of the electrochemical processes involved in these materials, X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and 57Fe Mössbauer spectroscopy at room temperature were coupled and performed on the as-prepared glasses and on ex-situ (after cycling) materials at different electrochemical states of charge. This new study will bring significant elements to optimize both the glass composition and its elaboration conditions to obtain high performance cathode materials without critical materials.

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Keywords: Oxide Glasses, Electrical Properties, Lithium Ion Batteries

Characterization and photo-viscous changes in phosphate-based glasses doped by copper ions

Jan Smolík *† [1], Zuzana Zmrhalová [1], Zdeněk Černošek [2], Eva Černošková [2], Ekaterina Samsonova [2], Petr Knotek [2], Jiří Schwarz [2]

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Phosphate glasses represent a significant group of phosphorus and oxygen-containing glasses with several potential technological applications. These materials exhibit relatively high values of coefficient of thermal expansion with low glass transition temperature and other processing temperatures which is interesting for sealing (1). Some phosphate glasses with suitable chemical compositions, e.g. calcium phosphate glasses, are also applicable in the field of biomedicine (2). In addition, they also have the short wavelength absorption edge typically located in UV region and are able to incorporate into their structure a relatively high amount of transition metal ions (1,3). These properties make phosphate glasses promising for several hi-tech applications, such as solid-state lasers (4), low-frequency optical fibers (5) or optical data storage (6). The advantage of these materials from the point of view of sustainable development is also the potential immobilization of radioactive wastes (7).
Copper can exist in glassy structures in several different chemical forms, i.e. metallic Cu(0), Cu(+) or Cu(2+) ions with various coordinations, which differently determine/affect the materials' properties, e.g. optical characteristics (8). The chemical form of copper in glasses is subsequently given mainly by the chemical compositions of starting materials, which react together during the synthesis, and applied experimental conditions (8-10).

In this work, we focus on the substitution of Zn(2+) ions by Cu(n+) ions (where n = 1 or 2) in the phosphate glasses and the influence of this substitution on their structure and properties, i.e. optical and thermal properties including also photo-viscous changes. All bulk materials (i.e. with 0-11.6 mol% of starting CuO) were synthesized by classical melt-quenching technique in a corundum crucible.

The obtained bulk glasses were chemically stable in the ambient atmosphere. The amorphous character or the presence of crystalline phase in glasses was detected using X-ray diffraction analysis. The real chemical composition of prepared materials was checked employing X-ray fluorescence spectroscopy. The information on the structure of the anionic part of glasses, i.e. the present phosphate-based structural units, was obtained using two different methods: Raman and 31P nuclear magnetic resonance spectroscopy. The characterization of incorporated Cu(n+) ions (oxidation state, coordination) was performed by electron paramagnetic resonance spectroscopy. The effect of Cu(n+) ions on the optical properties was examined using UV/Vis spectroscopy in the range of λ = 300-900 nm. Glass transition temperature of prepared mate-

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rials was determined by thermomechanical analysis.

The photo-viscous changes were examined similarly as in our previous work (11) employing a specially modified thermomechanical analyzer (TMA) with an optical transparent path (indenter) for *in-situ* laser illumination during the measurements. In this work, the photo-viscous changes were studied using two different approaches. The first approach is based on the measurement of the rate of indenter penetration into the sample (the change of penetrating indenter volume per time in the given time interval) during the isothermal measurements. The penetration rates for measurements with and without illumination were subsequently compared. In the second case, TMA was used for the common dilatometric measurements without and with illumination. For each illumination, solid state continuous-wave laser emitting at 808 nm (maximal laser power density incident on the samples ≈ 5.9 W/cm2) was used.

In this work, it was found that copper ions significantly affect the optical properties in the visible region due to the formation of a broad absorption band with maximum around 800 nm which intensity increases with the increasing amount of starting CuO. The values of glass transition temperatures have non-monotonous behavior affected by the amount of CuO and the structure of prepared materials. Illumination of the samples by 808 nm during penetration measurements at the same temperatures led to an increase in the penetration rate compared to that obtained without illumination. Based on the shift of penetration rates between illuminated and non-illuminated measurements, the local non-uniform overheating of the sample with ≈ 0.7 mol% of CuO induced by illumination is by ≈ 22 ◦C. Similarly, illumination causes the "apparent" decrease of glass transition temperature value compared to the dilatometric measurement without illumination which is for the same sample (0.7 mol% CuO) ≈ 20 ◦C corresponding well with the results obtained by penetration method. In addition, the magnitude of the difference between glass transition temperature without illumination and "apparent" glass transition temperature due to illumination increases with the increasing CuO content. The cause is probably higher overheating of materials as a result of the increasing absorbance at 808 nm associated with the increase of Cu(2+) ions content.

We believe that the results of this work could bring new findings from the point of view of the laser-solid state interactions and could help to determine the suitable Cu(2+) ions concentration for the successful laser micro-structuring (i.e. concave or convex microlenses formation) by synergy effect of the heat and continuous-wave laser in the visible part of the spectra. The laser micro-structuring of materials presents an interesting way for miniaturization of various devices.

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Keywords: phosphate glasses, copper ions, structure, optical properties, photoviscous changes

Characterization of Ag nanoparticles in heavy metal oxide glasses

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Metallic nanoparticles embedded in various glassy matrices are intensively studied due to their ability to modify the physical and chemical properties of the bulk glass phase. These nanomaterials dispersed in glasses find several applications, f. e. in ultra-fast non-linear photonics (e.g. optical and electro-optic modulators, switches, ultra-fast time response optical sensors), plasmonics and telecommunications. Silver nanoparticles attract a great attention due to their electrical, optical, catalytic and biochemical properties. Therefore, the researchers have focused on studying their physical, chemical, electrical, and optical properties (1, 2). Presence of metal silver nanoparticles in the glass matrix results also in unique linear and non-linear optical properties due to strong surface plasmon resonance. Non-linear properties of such nanocomposite materials depend mainly on the size, shape, distribution and concentration of metal nanoparticles (2).

The formation of silver nanoparticles has already been investigated in common glass systems based on silica, phosphate and borate oxides. However, these glasses exhibit much lower non-linear (Kerr) EO coefficient and also their transparency in infrared region is limited in comparison to the glasses based on heavy metal oxides. The heavy metal oxide glasses are further characterized by high refractive index, they show high stability against crystallization and are easy to prepare. They are characterized by high content of heavy metal oxides, usually PbO or Bi2O3, in combination with WO3, Ga2O3, GeO2 and/or other oxides. The PbO-Bi2O3-Ga2O3 system forms homogeneous thermally stable glasses with very good transmittance going from the visible up to mid-infrared region (up to 8μm). These glasses exhibit also better physical and chemical properties (higher chemical resistance, lower thermal expansion coefficient) in comparison with halide and other non-oxide glasses that are transparent in similar spectral region (2).

In this work the silver nanoparticles were formed during thermal treatment in glasses modified by Ag2O and Sb2O3 addition. These glasses were prepared by conventional melt-quenching in PtRh crucible in an electric furnace. The presence, chemical composition and structure of the silver metal nanoparticles were confirmed with transmission electron microscopy (TEM)

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and x-ray photoelectron spectroscopy (XPS) analysis. TEM confirmed an increased number of spherical nanoparticles caused by additional heat treatment. The indexes of the selected area electron diffraction (SAED) pattern recorded on such particles matched the fcc-Ag crystalline phase, high resolution (HRTEM) mode identified the periodical arrangement in the particles corresponding to d-spacing of (111) planes in Ag and the elemental state of the present Ag in the heat-treated sample was confirmed also by XPS. Electrical measurements were chosen due to their sensitivity to any changes in the material structure. The electrical conductivity of glasses containing Ag/Sb changed during thermal treatment (the investigated temperature range of 240–290 ∘C), while the properties of the base glass without Ag/Sb addition remained practically unaffected (3).

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Keywords: Keywords: silver nanoparticles, heavy metal oxide glass, PbO–Bi2O3–Ga2O3 glass system

Composition Design for New Generation Controlled Release Glass Fertilizer

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The rapid growth of the global population is leading to a decreasing amount of arable land per person. Consequently, to meet the escalating food demands of this expanding population, the use of fertilizers in agriculture has become unavoidable. Even though the chemical fertilizers are widely employed in order to increase the agricultural yield, they pose harmful effects on the environment and living organisms in addition to their high solubility in water results in a significant portion of nutrients being washed away from the soil before the plant uptake. Commercially available Controlled Release Fertilizers (CRFs) emerge as eco-friendly alternatives but face challenges in competing with chemical fertilizers. In the scope of ongoing research for novel CRF technologies, glass fertilizers have gained attention due to their rich nutrient contents, eco-friendly behavior and the controllable dissolution rates through compositional changes. In this study; various phosphate based glasses have been synthesized and investigated in terms of their physical, thermal and structural properties along with their chemical durability in different mediums. Compositional dependence on chemical durability of phosphate glasses and their potential to be used as agricultural fertilizers has been evaluated.

Keywords: Glass fertilizers, phosphate glasses, dissolution behavior, controlled release fertilizers

*Speaker

Chlorine solubility and speciation in Low Activity Waste nuclear glass synthesised under high pressure and different redox conditions

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Among the radioactive waste produced in nuclear power plants, the radioisotope chlorine 36 poses a serious threat for the public health and the environment due to its high mobility in the environment and its long half-life (301ky). The volatile character of Cl strongly limits the use of classical melt-quenching to form nuclear-waste glass. To propose a more efficient and durable solution for the immobilisation of halogens radioisotopes, a method using high-pressure has been proposed and shows strong increase in the Cl incorporation. Whereas the effect of glass composition and intensive conditions on Cl incorporation are fairly well-constrained, the effect of various oxidation conditions are partially known. Recent work suggested a decrease in Cl solubility with increasing oxidation conditions in discrepancy with recent results for iodine showing the opposite behaviour. In contrast with previous results, we found that the increase in oxidation conditions does not decrease the Cl solubility but instead slightly increases Cl solubility with perchlorate initial source.

The mechanism behind this behaviour is not yet clear as Cl speciation does not appear to change to an oxidized species. Therefore, a better understanding of the dissolution mechanism is required under various oxidizing conditions to formulate a semi-empirical model for Cl behaviour in nuclear waste glasses.

Glasses have been synthetized under high-temperature (1250◦C) and high-pressure conditions (1.0 GPa) with the following compositions: 65 mol.% SiO2, 5 Al2O3, 10 to 15 B2O3, 7 to 13 CaO and 5 to 22 Na2O. Two glass series were prepared with varying Cl sources that mimic different oxidation conditions: NaClO4, NaClO3 and NaCl between 9 to 16.8 mol.%. The Cl content after experiment reaches up to 6.5 mol.%. To characterize the redox and the structural environment of the Cl in the glasses synthetized, Cl K-edge X-ray Absorption Spectroscopy (XAS) and Cl 2p X-ray Photoelectron Spectroscopy (XPS) have been used.

The investigation by XAS of Cl k-edge showed X-ray Absorption Near Edge Structure (XANES) spectra dominated by Na-Cl and/or Ca-Cl signals with a threshold at 2823.8 eV and Emax at 2826.1 eV respectively, in agreement with previous work. The Extended X-ray Absorption Fine

*Speaker

Structure (EXAFS) spectra confirm the structural environment of the Cl- in glasses charge compensated by Na+ and/or Ca2+ cations at around 2.8 Å. Moreover, the EXAFS revealed the presence of a short local bonding around 1.7 Å that can be explained by the presence of Si-Cl bonds around 1.6 Å or Cl-Cl bonds at 1.85 Å. Cl 2p XPS results show a peak at _˜198.5 eV which can be attributed to Cl- species; no peak for the Cl oxidized species (i.e. ClO3- and ClO4-) are present. Although the spectra can be simulated with a single Cl- in agreement with previous work, we reconsidered the simulation of the Cl 2p XPS spectra in regards of our XAS results. Those simulations showed position of the bonding in agreement with previous studies for alkali/alkaline-earth cations bonded to Cl- and Si-Cl.

Keywords: Chlorine, X, ray Absorption Spectroscopy, X, ray Photoelectron Spectroscopy, Nuclear waste, Glass, high, pressure

Colour, composition, and rheology of obsidian

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Obsidian is the most spectacular natural lava and probably the oldest glass used by mankind for almost 10,000 years. After MORB, they are the most important natural glass on Earth in terms of quantity, and their formation is still the subject of much debate. As a glass, it is characterised by high silica (> 68%wt), alumina (> 10%wt), ferrous and ferric oxide content (1-5%wt) as well as the incorporation of significant amounts of intrinsic water. Although most widely recognizable in its black form, obsidian can occur in a broad range of colours and varying levels of opacity; it can exhibit significant heterogeneities in terms of composition and colour, and it is not uncommon for crystals to be incorporated within the glassy network.
Although the most common colour, black, is attributed to iron nano-inclusions within the glass, the exact nature of colouration in other types of obsidian is not well understood. Spectroscopic and nanoparticle analytical techniques may be able to reveal it. Due to its high water content, obsidian cannot be easily manipulated under heat, as common glass can be. However, it can be mixed with common glass to overcome this limitation. Viscometric techniques should provide insight into the differences in rheological properties among various obsidian types and offer an effective method to reusing it in the creation of modern artefacts.

Keywords: Obsidian, natural glass, colour, viscometry

*Speaker

Deciphering Glass Dissolution Rates: A Machine Learning Perspective for Prediction and Interpretation

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Understanding the long-term chemical resistance of glass is vital for safely immobilizing nuclear waste. However, predicting the dissolution rate of glass, which depends on factors like composition, pH, and temperature, is complex due to their intricate and nonlinear relationship. To address this challenge, we curated a comprehensive dataset comprising around 2100 glass compositions along with corresponding pH and temperature values. Further, we employed various machine learning models, including Artificial Neural Network and Xg-Boost, to predict glass dissolution rates. Results show that the Xg-Boost model performs reasonably well, achieving a training score R2 value exceeding 0.9 and accurately predicting dissolution rates. Furthermore, we explored the dependency of dissolution rates on composition, pH, and temperature using a game theory-based SHAP approach. Additionally, we transformed the composition into different descriptors to develop a universal model capable of predicting dissolution rates for compositions not present in the training dataset. Our results demonstrate that descriptor-based models perform comparably well to composition-based models, suggesting their potential as a generalized prediction tool for dissolution rates. Finally, we derived the functional form of dissolution rates in terms of various descriptors through symbolic regression, enabling the calculation of dissolution rates for any oxide glass compositions. Overall, this study sheds light on glass dissolution rates and facilitates the discovery of novel glass compositions for effective nuclear waste management.

Keywords: Nuclear Waste, Machine Learning, Glass

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Crystal growth in chalcogenide glass-formes prepared in different forms and its relation to viscosity and diffusion

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Chalcogenide glass-forming materials prepared in different forms (bulks, fibers, thin films) are very promising materials widely used in practice (e.g. lenses, fibers, filters, diffractive optical elements, diffractive optical elements, filters, optical recording discs for data storage, etc.). Optical and electrical differences between amorphous and crystalline phases of some chalcogenide systems and rapid and reversible switching between these phases are fundamental for using chalcogenide materials in data storage applications.

Knowledge of viscosity behavior, crystal growth phenomenon, and diffusion is important in producing, processing, and applying amorphous solids prepared in different forms (bulk glasses, thin films). This work focuses on crystal growth in Ge25Se75 bulk glasses and thin films prepared by thermal evaporation and solution processing. Crystal growth data were obtained using infrared microscopy measurements, showing a change in crystal morphology within the broad studied temperature region (250 – 550 ◦C). Nevertheless, a single crystal growth model could describe the crystal growth rates. The combination of viscosity and crystal growth data provides an extensive collection essential for crystal growth description in a wide temperature range. The found crystal growth model describing the experimental data provides information about the size of structural units incorporated into the growing crystals. The structural unit size is then used for the estimation of self-diffusion coefficients (D) that show a similar relation with crystal growth rates (u) as was found in molecular glasses (u ≈ Dˆ0.87).

Acknowledgments

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Keywords: crystal growth, chalcogenide glass, thin films

*Speaker

Development of glass formulations for sealing applications in high-temperature steam electrolysis

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Hydrogen production by high-temperature steam electrolysis is achieved using complex structures such as Solid Oxide Cells (SOC) stacks. Ensuring the sealing of this metallic/ceramic multilayer is technically very challenging. The sealing solution needs to meet several properties. Specific thermomechanical, chemical and electrical properties are mandatory for the sealing material in SOC stacks. Glasses and glass-ceramics have been identified as suitable candidates..

This study consists in the development of glass sealants for high-temperature electrolysis applications. Four glass systems have been identified as potential sealants for SOC currently developed at CEA. Based on literature review, glasses compositions were chosen due to their matching properties with the required ones.

Modifications of glasses compositions were studied in order to tailor sealants thermomechanical properties (glass transition temperature, softening temperature and Thermal Expansion Coefficient) and to reduce the probability for deleterious devitrification in SOC working conditions. Several samples have been elaborated and their properties characterized as raw and thermally treated glasses. Glass samples were thermally treated at different temperatures and for different durations. They were then characterized by SEM-EDS and X-Rays Diffraction to get insights into the crystallized phases. Thermal Expansion Coefficient of resulting glass-ceramic samples were also measured in order to assess thermal treatments influence.
This poster presents results obtained on a specific glass system.

Keywords: High, temperature electrolysis, Glass sealant, Thermomechanical properties

*Speaker

Effect of glass alteration on silver speciation in a phase separated glass

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Diffusion on glass material plays a key role in industrial and geological processes. Interdiffusion mechanisms are at the core of ion exchange processes, notably employed for modifying the surface properties of glasses (mechanical reinforcement, gradient in refractive index, glass coloration...). One of the challenges facing glass as a material for a sustainable future is a better control and use of these processes to reach new glass properties and improve their life cycle.
Many models and mechanisms have been developed and gives us a better understanding on how diffusion works. However, few experimental studies have been reported so far on the diffusion of ions in a heterogeneous media. The present study investigates how heterogeneities in composition impact the interdiffusion of metallic ions.

The present work consists in investigating the diffusion mechanisms in a borosilicate glass separated in two phases: a silica-rich one and a sodium-boron-rich one. Firstly, we explore the diffusion of silver ions through ion exchange, employing the salt bath method. Silver is mainly present in the boron-sodium rich phase, which is highly sensitive to the ambient humidity. Interaction between the silver-doped glass and ambient humidity leads to structural changes mainly in the boron-sodium rich phase: the silver, mainly in ionic form after diffusion, tends to precipitate into metallic nanoparticles through time. Experiments have shown that the size and distribution of the nanoparticles depend on the initial phase separation pattern, the conditions of storage and the ionic exchange conditions.
Our work mainly focuses on understanding how the differences in composition, chemical and physical properties between the two phases impact the diffusion of silver metallic ions, as well as their reduction and precipitation in nanoparticles.

Keywords: Phase separation, Glass Alteration, Silver, Nanoparticles

*Speaker

Extrusion of optical fiber preform – case of ZBLAN glass.

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ZBLAN glass name derives from its components: **Z**rF4, **B**aF2, **L**aF3, **A**lF3, **N**aF. Drawing optical fibers from ZBLAN glass has been of interest, because of the glass's unique properties, such as high transparency in a wide spectrum, low attenuation, low dispersion, and low refractive index. All these parameters make it a great material for multiple applications such as fiber sensors, lasers, lenses fabrication, supercontinuum light source generation, and others.
However, working with ZBLAN glass is challenging. ZBLAN glasses are prone to crystallization during multiple thermal treatments, which worsens the optical properties of the glass. This is due to the small window between the glass transition temperature (Tg) and crystallization temperature (Tc) peak. Moreover, the preparation of preforms by mechanical processing means is problematic due to ZBLAN glass brittleness. Mechanical processing also causes high material loss, which is unwanted taking into consideration the production cost and high-purity raw materials used.

The extrusion process of the ZBLAN glass for the preform preparation can overcome all the problems mentioned above. The general idea of the extrusion is to insert the glass into a steel mouthpiece and put it under pressure applied by a ceramic pushrod. The whole arrangement is placed in the furnace and precisely heated to a temperature a little above Tg and not reaching Tc. Then glass bulk is extruded to the desired shape. As a result, glass fiber preforms in the form of tubes and rods can be obtained, depending on the geometry of the mouthpiece. In the process, almost all the glass is extruded (only a small amount of glass leaves in the steel mouthpiece) allowing to avoid all the issues of grinding and polishing.

The aim of this research was to obtain ZBLAN optical preform with extrusion process and optical fiber assembled by rod-in-tube method from obtained components. The tube was extruded by the process described above; the rod for the core was prepared by grinding and polishing a ZBLAN glass doped with 2% PbF2. The preform was then drawn on the optical fiber tower. The resulting fiber was multimodal, of step-index profile with 200 μm outer and 100 μm core diameter. Analysis of the fiber included scanning electron microscopy (SEM) of fiber cross-section, measurements of fiber losses, and numerical aperture.

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Keywords: glass fiber, modeling, glass extrusion, ZBLAN

Fabrication of Magnetic Iron Oxide Nanoparticle Doped Phosphate Glasses

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The inert, transparent, and amorphous nature of glasses has highlighted these materials as promising candidates for the fabrication of novel multi-functional materials. Doping glasses with nanoparticles has the potential to design new materials where the advantages of a glass matrix can be maintained, while adding the functionality of nanocrystals. In order to achieve such materials, the challenges are (i) prepare functional nanocrystals suitable for doping into a glass, (ii) coat the nanocrystals to provide a protective and wetting layer to facilitate doping, and (iii) carry out glass making using low melting temperature glasses to preserve the coated nanocrystals, and improving sustainability. In this study, superparamagnetic iron oxide (Fe3O4) nanoparticles were synthesised using a co-precipitation method, due to its high yield and use of inexpensive materials. The particles were coated in a silica shell via a 'sol-gel' method, using sodium silicate as a silica source, a cheaper alternative to silicon alkoxides. Both bare and coated nanoparticles are magnetic and their powder XRD analysis displayed characteristic Fe3O4 peaks, with coated samples also showing a broad halo in the region of 20-30$\circ$ 2
theta, corresponding to the presence of amorphous silica. The average size of the Fe3O4 nanocrystals was calculated with the Scherrer equation, showing nanocrystals to be in the 13 nm range, in both bare and coated samples. TEM imaging showed particles were spherical in shape for both types of sample. Phosphate based 45P2O5-10Ca-45Na2O glass, with a low melting temperature, was synthesised via a melt quenching process. XRD analysis showed no crystalline peaks, confirming the amorphous glass structure. The incorporation of the nanoparticles into a glass matrix was attempted by remelting mixtures of nanoparticles and glass matrix. In order to maintain the favourable properties of both materials, preventing the dissolution of iron oxide particles, which could alter both the glass structure and nanoparticle functionality, is needed. A balance between the nanoparticle to glass ratio is vital. High doping levels could influence both the optical transparency, and amorphous structure of the glass matrix, while low levels may diminish the superparamagnetic properties of the nanoparticles within the glass. Methods to achieve a homogeneous dispersion of nanoparticles within the glass are also being explored.

Keywords: Multi functional material, low melting temperature, phosphate glasses, nanocrystal, doping

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Manipulating Crystallization Via ZrO2 Control in Na2O-Al2O3-SiO2 Transparent Nanocrystalline Glass-ceramic

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How to prepare excellent NAS transparent nanocrystalline glass-ceramic remains a challenge, in terms of transmittance, bulk sample, mechanical properties, and other aspects. In this study, we used P2O5 and ZrO2 as mixed nucleating agents, focusing on studying the crystallization process. The presence of ZrO2 separates the crystallization of nepheline and carnegieite crystals. At the same time, a diffusion barrier was formed and the crystal size was limited. We have prepared transparent nanocrystalline glass-ceramics with high optical transmittance of 91%. The hardness of glass-ceramic has a significant increase of compared to untreated glass, increased from 5.18 GPa to 7.15 GPa (increased by 38%). By regulating the crystallization of NAS glass through Zr, the crystal type and size were effectively controlled, which is prerequisite for high mechanical properties and transparency. We hope that this work can provide new ideas and methods for the preparation of transparent NAS glass-ceramics.

Keywords: Transparent glass ceramic, Crystallization

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VITRIFICATION OF WASTE ARISING FROM DISMANTLING OPERATIONS USING DEM&MELT TECHNOLOGY

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Dismantling operations of end-of-life nuclear facilities produces, or will produce, various new waste, mostly of lower activity than fission products, among which significant volumes of ILW-LL – long lived intermediate level activity waste, which will need to be managed. The nature of the waste is very wide-ranged, compared to the waste usually vitrified, in terms of chemical composition and physical form (solid deposits, sludge or liquid solution), depending on their origin and according to the mode of recovery or storage of this waste during dismantling and decommissioning operations.
Among them, sludges/slurries represent a significant part of the produced waste. Their chemical composition and their humidity level, which can be variable, make their conditioning a challenge. One way to manage this waste is the vitrification process. This process has the advantage of stabilizing the waste in an inert mineral matrix, and of reducing the volume of waste to be stored, thanks to the high waste incorporation capacity of glass. In order to significantly increase the waste loading, a crystallized matrix or a composite matrix can be considered.

The In-Can vitrification tool DEM&MELT, developed by the consortium CEA (The Alternative Energies and Atomic Energy Commission), Orano and ECM Technologies is flexible enough to accommodate a varied waste stream and particularly adapted to sludges/slurries from dismantling operations. For this purpose, it is important to develop a vitrification adjuvant with optimized composition, in a suitable form in accordance with feeding system options.

Examples on inactive surrogates slurries from Fukushima Effluent Treatment* and on a sulphate-rich-sludge will be described in this study. If the first example has been investigated from the Laboratory to Pilot scale; for the second one, the temperature should be lower than usual ones used for glass elaboration, due to the composition of the waste, involving the use of low-viscosity frits. In this case, only lab scale route has been explored up to date.

Thanks to a panel of characterization methods such as differential thermal analysis (DTA), thermogravimetric analysis (TGA), X-ray diffraction (XRD), scanning electron microscopy (SEM), chemical analysis and viscosity measurements, precursors and wasteforms have been analysed, in terms of microstructure, durability, waste-frit reactivity and some adjuvants have been selected as promising precursors. Thanks to these results, the formulation of the different adjuvants will be optimized and tested on different sludges. The pilot scale extrapolation and the sustainability of the wasteform over time will then be explored.

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**Fukushima Effluent Treatment Waste* refers to the secondary wastes generated from the decontamination systems for contaminated water in Fukushima Daiichi Nuclear Power Station.

Keywords: Vitrification, In, Can Melter, sulphate, rich sludge, low, viscosity glass frit

Noble Metal Nanostructures Based Surface-enhanced Raman Scattering Fiber Probe for Trace Molecular Detection

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The detection performance of SERS fiber probes is highly determined by the geometries, morphologies, and components of noble metal nanostructures. In this research, different Ag, Au and alloy nanostructures were synthesized through chemical reduction method. The nanostructures were homogeneously coated on glass fiber end-tip for SERS fiber fabrication. The compositions, shapes, and sizes distributions of nanostructures were analyzed by XRD, TEM, and EDS.The stronger and larger areas of LSPR induced electromagnetic field from nanorod dimer and carbon dots decorated Ag nanoparticles implied better SERS behaviour. The lowest detection limit can reach 10-11 M for Au/Ag alloy nanorod fiber probes (Fig. 1 a), and 10-9 M for carbon dots decorated Ag nanoparticles fiber probes, using CV or R6G as an analyte. The relationship between Raman intensities and analyte concentrations showed well linear, for quantitative analysis, and the fiber probes showed excellent reproducibility and stability. Furthermore, carbon dots decorated Ag nanoparticles fiber probes were applied for trace Hg2+ ions detection, ranging from 10-5 to 10-11 M (Fig. 1b), from carbon dots reduced Hg meta surface wrapping on Ag nanoparticle surface, which decreased Raman intensities from CV analyte. The fiber probes can be applied for low-content chemical, biological molecules, and other detection.

Keywords: Surface, enhanced Raman scattering, Noble metal nanoparticles, Optical fiber, LSPR

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From Solid Inclusions to Sustainable Glass: Understanding Alumina Dissolution

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Commercial glass manufacturing faces the challenge of avoiding the presence of solid (mineral) inclusions that affect mechanical, processing, and aesthetic properties. The development of sustainable glass technologies that reduce primary (raw materials) and or secondary (heating process) CO2 emissions must comply with quality constraints and avoid mineral defects originating from refractory phases.
This problem requires a thorough understanding of the dissolution kinetics of typical refractory phases such as alumina in the context of an industrial furnace. Existing studies on alumina corrosion offer empirical laws, but their application to real-world problems remains challenging. At the scale of glass melting furnaces Computational Fluid Dynamics (CFD) is a promising tool but requires precise physicochemical data to complete quality index submodels.

In this context, alumina dissolution in an industrial soda-lime-silica glass melt has been studied at different temperatures of interest for glass fusion in static conditions (without convection). Employing a cylindrical slab approach inspired by methods developed in the geoscience literature, the effects of critical variables are isolated, bridging the gap to industrial applications.

The experimental setup involves stacking glass and alumina disks, encapsulated in a platinum-gold foil, subjected to temperatures ranging from 1300 to 1450 ◦C. Quenching and subsequent analysis through scanning electron microscopy (SEM) and electron probe microanalysis (EPMA) provide insights into dissolution behavior.

Glass compositional profiles exhibit uniform behavior across the studied temperature range, suggesting a diffusive control mechanism. On the other hand, strong coupling between elements is observed requiring the use of a multi-component diffusion approach to model alumina dissolution.

Eigenvalue analysis of diffusion matrices reveals two distinct exchange reactions: alumina with alkaline earth oxides and alumina with silica and alkalis. Activation energy calculations show Arrhenian behavior, offering insights into underlying energetics.

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Combining our results with literature data for alumina dissolution for a range of melt compositions and temperatures, we find that the effective binary coefficient of diffusion (EBCD) for alumina is very well correlated with viscosity, indicating an important control of atomic mobility in the liquid that may be related to Eyring-type diffusivity of networking-forming ions.

In summary, this study unravels mechanisms governing alumina dissolution in industrial glass melts, providing essential insights for optimizing glass quality. The findings contribute to the understanding of dissolution kinetics, refractories corrosion, and offer a basis for optimizing glass manufacturing processes for a more sustainable future, in the context of current incentives and the urgent need to use alternative raw materials and increase use of cullet, whose composition can be more variable than current raw materials.

Keywords: dissolution, alumina, digestion, diffusion, soda, lime silica glass

Fibrous glass electrodes for all solid-state batteries

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In recent years there is notable advance in rechargeable energy storage devices driven by geopolitically forced decline from fossil fuels. Main focus is on lithium-ion batteries utilized in almost all aspects of human life ranging from small electronic devices and EPS to electric transportation vehicles. However prevalent construction designs have several problematic areas. High price, high weight, relatively short lifetime under high power drain and most importantly safety. They contain highly flammable electrolytes, toxic materials and in case of thermal runaway release toxic and corrosive fumes. These problems can be addressed by utilizing all solid-state battery systems. Major drawbacks are generally solid materials low conductivities, therefor best solution is using thin layer materials ranging from tens of nanometres to several micrometres. There are several promising new materials especially silicon to be used as anode with reversible capacity almost 10 times higher than currently utilized graphite electrodes. Main issue with silicone is high expansion levels during lithiation, almost 300%. This again can be however addressed by transferring to submicron or nano dimensions.
Also, large number of solid electrolytes have been developed, where several high ion conductive types of glass can even surpass bulk polymer materials.
Very convenient method of producing thin-sheet nanomaterials is electrospinning. Thaks to recent development in upscaling production, it has become viable method to produce materials with electrochemical purity in amounts sufficient for industrial production unlike other nanomaterial production methods. It has been reported that it is possible to obtain fibres as thin as 2 nm in diameter. It is possible to prepare complex materials through sol-gel methods, however introducing highly active particles into spinning solutions has proven to be more convenient and more importantly cheaper option. It also allows to use recycled materials or even byproduct unused earlier. There are however several major issues. Spinning solutions, a processing conditions must be optimized to supress particle agglomeration and sedimentation, premature condensation, even particle distribution, and reaching uniform fibre distribution.

This study is focused on characterising morphology and properties based on processing and postprocessing conditions with 29Si NMR, XPS, XRD, EIS, laser confocal and electron microscopy. For this study glassfibre thin-sheet mats via sol-gel based on SiO2 filled with recycled active materials, were prepared from TEOS and PVP mixtures. PEMs were separated from EOL EV batteries, silicon from EOL monocrystalline solar cells. Next step of production is high pressure compacting followed by high temperature calcination in controlled atmosphere. Produced fibres retain its flexibility and functionality even after short exposure up to 1000∘C. Based on material testing, this technology seems promising in creating safer, energy and cost-effective batteries for EV application.

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Keywords: electromobility, recyclation, battery, glassfiber, nanofiber

Investigation of the structure of sodium borates and silicoborates at high temperature in both solid and molten states using high-temperature NMR

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Boron-containing glasses exhibit extensive applications in various fields, such as temperature- and chemical-resistant containers, fiber composites, optical components, display screens, and bioactive materials. The properties of these glasses are predominantly governed by the structures of their liquid precursors, which undergo increasing order during cooling and solidify during the transition to glass. The dynamics of this structural evolution are particularly intriguing as they influence the location of the transition and are intricately linked to viscosity and diffusivity. Given that microscopic processes dictate the macroscopic properties of these materials, obtaining atomic-scale information on both structure and dynamics with changing temperature is imperative. This study focuses on investigating the structural properties of various boron glasses at elevated temperatures. High-temperature nuclear magnetic resonance (HT NMR) (see Fig.1) and magic angle spinning (MAS) NMR techniques were employed to analyze different compositions of sodium borates and silicoborates glasses. The high-temperature experiments were carried out under a nitrogen (N2) atmosphere, reaching temperatures up to 1100∘C. Utilizing the information from high-temperature NMR 11B spectra obtained at high magnetic field (17.6 T), we proposed structural models to establish a comprehensive framework describing the physical characteristics of these glasses. Fig. 1. High temperature NMR experimental setup

Keywords: glass, high temperature NMR, theory and methods, instrumentation, exotica

*Speaker

Luminescence of Nd3+-doped LaF3 glass-ceramics enhanced with Ag nanoparticles

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Rare earths (RE) doped transparent oxyfluoride glass-ceramics (GCs) have demonstrated to be promising materials for applications in different fields, such as solid-state laser, down and up conversion phosphors or optical amplifiers. These materials combine the low phonon energy (300- 500 cm-1) of fluoride nanocrystals, which are known to be efficient host for RE ions; with the good mechanical, chemical, and thermal properties of the oxide glasses (1-4).
The majority of applications using the luminescence of RE ions involve electronic transitions of RE ions between states within a *4f* configuration and the corresponding emission lines cover the entire optical spectrum, from UV (ultra-violet) to NIR (near-infrared). Despite the excellent and encouraging results obtained so far, the intrinsically forbidden-nature of the *4f-4f* transitions is reflected in quite low absorption and emission cross sections and this is an important and fundamental problem still unsolved. Several ways to increase the luminescence efficiency of RE-doped materials for different applications have been summarized recently (5-7). A promising alternative is the use of metallic nanoparticles, such as Ag and Au, to increase the local field strength around the RE ions through the surface plasmon resonance (SPR) produced at the surface of the metallic nanoparticles when excited by light (8,9). Some authors reported an improvement of the photoluminescence of almost 300% in RE-doped glass-ceramics after introducing noble metal nanoparticles (10).

Nd3+-doped, LaF3-based, transparent oxyfluoride glass-ceramics (GCs) containing Ag nanoparticles (NPs) have been prepared by a melt-quenching method. Different Ag-containing precursors were employed being AgNO3 the most suitable for obtaining Ag NPs segregated in the glassy matrix. Transmission electron microscopy and X-ray absorption spectroscopy determined the presence of Ag0 NPs, with higher concentrations in glass-ceramics produced in an inert atmosphere (N2).

The spectral features of emission and excitation spectra, in addition to the different lifetimes of the 4F3/2 state in the co-doped samples with Ag NPs, demonstrate the incorporation of Nd3+ ions into the LaF3 nanocrystals. The relative contribution to the emission from Nd3+ in the nanocrystals depends on the Ag concentration and atmosphere. The glass-ceramic co-doped

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with 0.2 wt.% Ag, thermally treated in N2, exhibits the highest contribution of emissions attributable to Nd3+ ions in the crystalline phase. The crystalline fraction is similar to that of an Ag-free sample synthesized under analogous conditions. This increase of the luminescence is, therefore, attributable to the enhancement of the local electric field of the LaF3 nanocrystals due to the presence of Ag0 NPs**.**

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Keywords: transparent glass, ceramics, fluorides, SPR, rare earth ions, metallic nanoparticles.

Rheology of partially crystallized simulated nuclear glass melts

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In France, high-level wastes (HLW) resulting from the reprocessing of spent Uranium OXide fuel are conditioned in sodium alumino-borosilicate glasses. The formulation of such glass results from a compromise between the waste-loading (waste mass to final glass mass ratio), the technological feasibility of the glass at an industrial scale, and its long-term behavior. To date, the compositions of the alumino-borosilicate glasses are formulated to avoid any crystallization in the melt. However, the tolerance of crystals in the glass melt could allow to reach higher waste loadings, which would decrease the number of glass-containers and therefore the cost of the long-term storage.
Previous studies have shown that the presence of particles alters some properties of the glass melt, particularly its rheology. This is the case of platinoids elements, which are poorly soluble in the glass matrix (solubility limit generally < 100 weight ppm), and which precipitate as RuO2 needles and PdTe beads. Hanotin et al. (1) observed that less than 1 vol% of platinoids particles leads to a very shear thinning behavior. Unlike platinoids particles, for which the volume fraction is constant during glass melting, the fraction of crystals considered in this study depends on temperature and time. Therefore, the study of the effect of crystals on the rheology of the melt has to take into account the time-temperature dependency of the crystal fraction. In addition, crystals morphologies are also likely to change with temperature (2). Two types of crystals observed in nuclear glasses due to exceeding solubility limits were chosen for this study: 1) cerianite, which generally shows a cubic morphology and slow crystallization kinetics 2) apatite, which generally has an acicular shape and fast crystallization kinetics (2). The morphology of crystal is expected to impact the rheology behavior of the melt (3).
In this work, we first evaluate the crystallization properties of both crystal types at high temperatures (around 1100◦C) in both static and dynamic conditions (absence / presence of flow). In static conditions, thermal treatments are performed in a tubular furnace. For the experiments in dynamic conditions, a stress-imposed rheometer with a multiblade rotor is used. The stirring is controlled by changing the imposed stress – the viscosity is acquired simultaneously. Post-mortem analyses are carried out for each condition in order to assess the microstructure of the frozen melt. For the studied cerianite fractions (up to ˜1 vol%), virtually no change in the viscosity is observed. For the apatite case, however, a very shear thinning behavior is observed, similar to that observed for platinoids particles in previous studies (figure 1). Rheological models are tested to describe the behavior of the suspension. These findings will be useful to simulate the behavior of the glass melt during the vitrification process. References

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Keywords: rheology, glass melt, crystallization

Visualizing the energy landscape of glassy systems

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Understanding the energy landscape is key to discovering glasses with targeted properties since the landscape encapsulates a system's complete thermodynamic and kinetic behavior, including its non-equilibrium properties, such as relaxation and metastable phases. However, the curse of dimensionality prohibits one from effectively visualizing the energy landscape-the energy landscape of an N-atom system has 3N dimensions. Here, we propose a method to visualize the complex energy landscape. We demonstrate that the proposed low-dimensional projection aligns well with the curvatures of the actual landscape, validated through Hessian analysis. Further, we show that we can gain interesting insights into the behavior of different gradient-based and machine-learned optimizers using the proposed visualization approach. Through this study, we aim to enhance comprehension of energy landscapes and contribute to a fundamental understanding of the physics underlying glassy materials. Furthermore, we anticipate that our method will expedite the discovery process of these materials.

Keywords: Energy Landscape, Visualization, Optimization

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Physico-chemical and structural transformations under shock of hydrated minerals

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My thesis is linked to the sustainability of hydrated glasses. The main objective is to study the fate of water contained in hydrated minerals under hypervelocity impact conditions. The methodology developed is established by studying silica glass, a material already referenced under impact (Thesis C. Dereure 2019). The focus here will be on the evolution of the material's density and variations in its hydration rate.

This implies many analyses of samples before and after impact experiments by characterization of hydration in depth, mostly with Raman Confocal Spectroscopy. For now, we are mostly focusing on the hydration process of glasses which can be obtained by diffusion and reaction of water in a high temperature and pressure atmosphere. The impact will be then produced by using the analogy between hypervelocity impact and laser-induced shock (Pirri 1977) and imply various national facilities like those of LULI laboratory (Laboratoire pour l'Utilisation des Lasers Intenses - UMR 7605 CNRS), equipped with in-situ diagnostics such as VISAR and umbroscopy.

The thesis is a part of the ANR SiCLAMEN.

Keywords: Silica glass, Choc, Water, Hydration, Sustainability, Silica glass, Hypervelocity impact, Mechanical properties

*Speaker

Phase separation inhibits crystallization of lithium aluminosilicate crystals

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The various characteristics of glass-ceramics are determined by the features of its precipitated crystals, and the formation of crystals will be affected by phase separation. However, characterizing the nanoscale phase separation and nucleation mechanisms of glass-ceramics has been a challenge. Here, we present direct evidence that glass phase separation can effectively inhibit crystallization. The nanoscale phase separation in the glass phase prior to formal nucleation and crystallization has been demonstrated by NMR. After the phase separation treatment, the crystallization is greatly inhibited. This phenomenon can be attributed to the preferential migration of O2- and Li+ ions towards the phosphorus-rich regions under high-intensity P5+ ion fields. Consequently, spontaneous phase separation occurs during the cooling process of the melt, leading to an increased degree of aggregation in the silicon-rich region of the glass network. We hope that this work can provide a new idea for controlled crystallization.

Keywords: Phase separationcrystallizatin glass, ceramic

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Viscosity measurements of As-Se chalcogenide glass and its use for determination of crystal growth kinetics

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Viscosity is a material property which defines how well the material flows. Its knowledge is very important, especially for glassy materials. The crystal growth kinetics is also influenced by viscosity. The diffusion of structural units to the crystal-glass interface is usually very hard or impossible to measure, but it can be replaced by the Stokes-Einstein equation, which correlates diffusion with viscosity. Also, the kinetic crystal growth models are viscosity-dependent, making the knowledge of rheological properties essential (1,2). Our study group, the Málek Research Group, have been interested in the viscosity measurement for a long time, and we've been trying to describe the viscosity of various noncrystalline materials in a broad range of temperatures, from the melted material and undercooled melts to amorphous glass. We've been using many different experimental techniques to achieve that, some of which are introduced in the presented study. The use of these techniques will be demonstrated on an As-Se glassy system.

The first method of viscosity measurement used to study the above-mentioned glass is the thermomechanical analysis (TMA). In this method, the material is being isothermally deformed by force with different setups. The first setup uses different kinds of indenters, where the penetration depth is measured, and from the difference in the height of the measuring probe in time, the viscosity value is calculated. The most used indenters have the shape of a hemisphere, which is suitable for viscosities in a range of 109 to 1013 Pa·s, and a cylindrical shape, which is ideal for a range of 107 to 1011 Pa·s. The parallel plates are another setup used for lower viscosities, mostly in a range of 105 to 107,5 Pa·s. It is performed by pressing a cylindrically shaped sample between two corundum plates, and the value of viscosity is determined from the change in the sample's height (3). All three of the above-elaborated setups with the proper formulas used to calculate viscosity (h) can be seen in Figure 1.

Another method that can be used to measure viscosity is nanoindentation. It is comparable with TMA in a similar execution since this method also uses an indenter, which penetrates the surface of the sample, and the viscosity is calculated from the depth of the indent. The main difference is that the penetration depth is much smaller compared to the TMA, being around tens of nanometres, which makes this method suitable even for measuring samples in the form of thin film. The formula to calculate viscosity from the nanoindentation experiment is then expressed in Figure 2 (5,6):

The final method used for viscosity measurement mentioned in this study is the Pressure-assisted

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melt-filling technique (PAMFT). This method is different from the previous ones because it measures the viscosity in melts. The sample needs to be in the form of a fibre, which is then put into a capillary of similar inner diameter. This capillary is then spliced with another capillary with a much smaller inner diameter, as is shown in Figure 3. This setup is heated above the melting temperature, and the melted sample is pushed from the larger to the smaller capillary with an inert gas. The distance the melt has flown is recorded in time. The experimental setup is shown in Figure 3, with the formula used to evaluate viscosity. This method is a promising new approach for measuring chalcogenide glasses because of their high volatility and corrosivity. Another advantage is using very small amounts of material (less than 0,1 mg). This method's resulting viscosities ranged from 10-1 to 102 Pa·s, but even smaller values could be reached (7,8).

Multiparametric phenomenological models express the temperature dependence of viscosity. One of the simplest models is the Arrhenius-type equation, which is shown in Figure 4 equation (a). Since the logarithmic form of this equation is linear, it is useful only for describing a short temperature range of viscosity data or for materials with low fragility. However, this model is not very useful for most materials (7). The Vogel-Fulcher-Tammann (VFT) equation (9), shown in Figure 4 equation (b), is more suitable for rheologically complex materials. The three-parameters model can describe material behaviour in a more detailed way. One of those parameters is the fragility (m) mentioned above, which is visualised by the curvature of the fitted model. Figure 4 equation (c) is the transcription of the Mauro-Yue-Ellison-Gupta-Allan (MYEGA) equation (10), described by three parameters and more suitable for semi-fragile materials. The latter model is also used to describe the viscosity data of As20Se80, shown in Figure 4, which were measured by all three above-mentioned experimental techniques (4).

The three mentioned viscosity measurement techniques will be shown in the poster that will be presented at the ICG Spring School 2024. The results of these measurements are demonstrated on AsxSe100-x chalcogenide glass of x = 5, 10, 15, and 20 compositions together with the viscosity model, which best describes their behaviour.

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Keywords: Viscosity, Glass, Kinetics, Chalcogenide

Topology Optimization for Structural Design of Glass: Numerical and Experimental Study

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The application of structural topology optimization to glass may enable the design of architectural and lightweight glass structures. There is still a lack of specific topology optimization tool for such a brittle material. Thus, the purpose of this work is to develop a custom numerical topology optimization for the design of wide-span glass structures. This allows to obtain monolithic load-bearing glass components which can have high strength-to-weight ratio. Here, we consider two volume minimization problems in which global displacement and global maximum principal stress are taken into account. The optimization algorithm is developed based on density method with robust filtering method and the Method of Moving Asymptote (MMA) is used as the standard optimizer. Two benchmark examples with parametric study of the algorithm are presented in both 2D and 3D design problems. We find that the custom topology optimization tool enabled not only to minimize the volume of the structure but also to increase the structural performance (stress distribution). Finally, validation experiments are performed on the topologically optimal structures for the MBB beam cut by abrasive waterjet.

Keywords: Topology optimization, lightweight glass structure, Failure behaviour

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Irradiation effects on the leaching of nuclear waste glasses: Understanding and modeling of leaching mechanisms

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In France, borosilicate glass, known as R7T7, is used to contain high-level, long-lived nuclear waste from spent fuel reprocessing (1). The glass canisters containing these wastes are destined to be stored in deep geological repository. After several hundred years, groundwater is expected to come in contact with the glass, which will have been self-irradiated by the radioactive elements it contains. Alteration of the glass by water, whose structure and properties will have been modified by the effects of it self-irradiation (2), represents the most important source of radionuclide dispersion in the environment.
Insofar as the alteration phenomenon takes place on a time scale that is inaccessible, modeling enables to simulate the very long-term glass behavior. In this case, the GRAAL 2 model (3), which will be used during this Ph.D. study, allows the prediction of the glass constituent elements releases in solution. Nevertheless, GRAAL 2 does not allow the distinction between the alteration of a pristine glass and a radioactive glass yet.

In order to better understand the alteration mechanisms of nuclear glasses and to adapt the GRAAL 2 parameters to radioactive glasses, this work focuses on the study of the alteration mechanisms of a simplified borosilicate glass, called ISG (International Simple Glass). Its simple chemical composition (SiO2, B2O3, Na2O, Al2O3, CaO, ZrO2) exacerbates some phenomena, such as leaching and irradiation-induced structural modifications, and makes characterization and modeling easier (4). In order to simulate the various irradiation sources of a radioactive glass, we will study two types of samples in parallel:

- Radioactive curium-doped ISG glass, which has accumulated electronic and nuclear doses for several years;

- Non-radioactive ISG glasses, which will be irradiated using external beams under different scenarios (electrons, He, Au, Xe, Si, Ne...) to simulate the effect of self-irradiation.
The methodology chosen to study the effects of irradiation on the structure and properties of

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the two types of glasses as well as their behavior under alteration by water will be presented and supplemented by the first results obtained. The stored energy of each sample depending on the irradiation scenario will be quantified using calorimetric studies carried out by DSC (Differential Scanning Calorimetry) (5), and different leaching protocols will be used to favor some alteration mechanisms over the others and thus independently measure the impact of irradiation on each of them.

Keywords: ISG, Leaching, Irradiation, modeling, curium

Molecular Dynamics Modelling of Speromagnetism in Fe4(P2O7)3 Iron Phosphate Glass

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The study uses molecular dynamics to generate multiple models of Fe4(P2O7)3 iron phosphate glass before assigning spin moments to each iron atom (fig. 1) to generate magnetic parameters for each structure. The study is based around the findings discussed in Al-Hasni et al (1), which describe the spin structure of Fe4(P2O7)3 iron phosphate glass as being "interpreted in terms of speromagnetic order". Interatomic potentials and Fe4(P2O7)3 crystal structure coordinates are input into DL_POLY molecular dynamics software to simulate the melt and quench steps needed to generate a glass model. Spin moments are then added to each iron atom, with these details input into Spinvert, a software designed to optimise paramagnetic spin distributions. Spinvert outputs diffuse neutron scattering curves, as well as data related to spin correlation. These can then be used to compare the modelled structures to experimental data to validate the legitimacy of each model. (1) B Al-Hasni, G Mountjoy. Structural investigation of iron phosphate glasses using molecular dynamics simulation, Journal of Non-Crystalline Solids, Volume 357, Issue 15, 2011

Keywords: Computational, Modelling, Magnetism, Iron, Phosphate

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Optimizing Oxynitride Glass: Exploration of Production Techniques and Properties

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Historically, oxynitride glasses have faced challenges due to relatively high production costs, impurities, and non-transparency in the visible region. Likely contributing factors include the rapid decomposition of the nitrogen source (SiN) at high temperatures and the formation of metal silicide during the melting process. The replacement of SiN with AlN, along with the use of very pure starting materials, impacts the rate of decomposition and the formation of metallic silicide, significantly influencing the final glass quality. This presentation will provide an overview of oxynitride glasses in the Ca-(Al)-Si-O-N system, focusing on their preparation using different techniques, variations in properties with nitrogen and modifier cations contents, and issues associated with developing fully transparent glasses.

Keywords: Oxynitride glass, nitrogen rich glasses, high calcium content, microhardness, refractive index.

*Speaker

Preparation of a silicon-based anode from nanofibres prepared by the electrospinning

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The rapid growth of electric vehicles (EVs) has become a prominent trend in the automotive industry, driven by advancements in lithium-ion batteries (LIBs). The anode, or negative electrode, plays a crucial role in battery performance, and the prevalent use of graphite electrodes is attributed to their high porosity, conductivity, low weight, and cost-effectiveness. Nevertheless, scientists have recently explored monocrystalline silicon as a promising alternative to graphite. This shift offers advantages such as higher capacity, increased energy density (both volumetric and gravimetric), and a generally recognized safety benefit due to silicon's non-flammable nature. However, challenges arise from silicon's pronounced expansion and contraction tendencies during battery cycling. In this study, we address this issue by employing silicon in the form of nanofibers, which exhibit greater resilience to changes during cycling, ensuring enhanced battery stability throughout its lifecycle. Monocrystalline silicon particles were utilized as the active component in the nanofibers, obtained through grinding and sieving. A solution comprising these particles, along with organic precursors (PVP and TEOS), was processed using the electrospinning method to form fibres. To remove the polymeric PVP component, the fibres were subsequently annealed at 650 ◦C.
The transformations in material properties before and after the final annealing were analysed using X-ray fluorescence analysis, and changes in the phase composition were identified through X-ray diffraction analysis. The characteristics of individual fibres were observed using scanning electron microscopy and optical confocal microscopy.

Each annealed layer was pressed to achieve the highest density of active material and maximize the contact between individual fibres. The pressed layers were also examined using optical confocal microscopy. Additionally, the electrochemical properties were measured on the anode material.

This study employed a comprehensive approach combining X-ray techniques, microscopy, and pressing methods to investigate the material changes and optimize the structure of annealed layers. The results provide valuable insights into the characteristics and interactions of the individual fibres in the context of their application in advanced materials and energy storage devices.
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Keywords: nanofibres, siliconbased anode, electrospinning, batteries

Subcritical crack growth of SiO2-B2O3-Na2O amorphous phase separated glasses

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Oxide glasses, commonly used in everyday life, have a major drawback: they have a brittle behavior. In a vacuum, abrupt failure occurs when the stress intensity factor (K) is greater than the fracture toughness (Kc). When exposed to the environment, small pre-existing flaws can grow even under relatively moderate stresses. This sub-critical crack growth is also commonly referred to as stress corrosion cracking (SCC).

Over the years, researchers have evidenced a clear dependence of crack velocity (v) as a function of K, with v depending on the temperature (T), relative humidity (RH) and chemical composition (CC) of the glass. Below the fracture toughness (Kc), three different regions have been identified, corresponding to different crack propagation mechanisms. Below a threshold limit, called the environmental limit, there is no crack propagation. In region I, the crack front velocity is controlled kinetically by the reaction between water and the stressed bonds at the crack tip (1). Crack velocity (v) data follow Wiederhorn's exponential law with an apparent activation energy (2). Data can also be fitted using Maugis power law. (3) Water diffuses towards the crack tip, and its time to reach the crack tip is the limiting factor in region II, leading to a plateau in the log(v) vs. K curve. The crack velocity increases exponentially again with K in region III, which ends once Kc is reached (1).

Several studies have been carried out on subcritical crack growth in oxide glasses, but this phenomenon is less known for phase-separated glasses (4). Recently W. Feng *et al.* studied amorphous phase separated (APS) SiO2-B2O3-Na2O glasses (4)(5). The objective of this work was to understand the influence of glass structure (S) on fracture properties. For this purpose, pristine glasses were compared to glasses of the same composition that went through different annealing protocols. These annealing protocols triggered secondary phase separation, greater than the rings. The size of demixed zones increases proportionally with the cubic root of the annealing time. How this secondary structure plays on the environmental limit and region I was investigated.

In our lab, we captured the v(T,H,CC,S) vs. K curves using double cleavage drilled compression (DCDC). Samples undergo SCC tests using a dual screw Deben machine in a well-controlled

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environment (T = 19 ± 1 °C ; RH = 40.0 ± 0.5 %). Crack growth is monitored by means of a tubular microscope and a LabVIEW program. Crack velocities (v) are obtained for region I and the environmental limit by post-processing images of the crack front position. Velocities correspond to a range between 10ˆ-11 and 10ˆ-5 m/s.

To understand the link between the structure and fracture properties, additional tests were required. Atomic Force Microscopy (AFM) was used for post-mortem analysis of the fracture surface of the DCDC samples to capture the secondary phase size. Optical observations, X-ray diffraction (XRD) and Nuclear Magnetic Resonance (NMR) and Raman spectroscopies were also carried out to characterize the glass structure.

The poster will concern these previous works and will highlight future endeavors.
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Keywords: stress corrosion cracking, fracture, glass, amorphous phase separation

Statistical methods and data-driven models to predict glass melt properties

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In situations where theoretical models cannot be efficiently applied to calculate glass properties, empirical statistical models are often required. It is typically the case when glass contains a high number of components. Since the end of the 19th century, it is known that under certain conditions, silicate glass properties can be expressed as a simple linear combination of oxide contents. This "Principle of Additivity" was initially introduced to calculate heat capacity of glass, before being extended during the 20th century to a larger number of properties: optical, thermal, mechanical or rheological properties. In the 1990s, American scientists from PNNL developed a statistical methodology to establish robust property-composition models applicable to the formulation of nuclear waste conditioning glass. Since the 2000s, significant increase in the power of computer tools has allowed to use highly efficient algorithms in the predictive methods of data mining. For example, glass transition temperature can be accurately predicted by using neural networks. Glass viscosity prediction is much more challenging because of huge variability of this property on temperature and composition scales. An innovative methodology recently developed by glass formulation scientists at CEA, with the support of Orano and EDF, combines statistical techniques of experimental designs, multilinear regression and neural networks. It uses glass formulation data generated at CEA over the past 30 years as well as large amount of data collected from the literature and from commercial database. Results obtained for glass transition temperature and viscosity predictions are very accurate, compared to other statistical models already published in the literature.

Keywords: Machine Learning, viscosity, prediction, database

*Speaker

Sol-gel derived aluminum doped zinc oxide (AZO) films

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Among transparent conducting oxides (TCOs), which are widely used in the design of electronic devices and solar cells due to their high transparency in visible light wavelengths and superior electrical conductivity(1), ZnO based ones are favored for their wide-bandgaps(2). Doping ZnO with Al, Ga, or In is an effective method for reducing its electrical resistivity(3). Despite the altered lattice structure of ZnO, AZO exhibits non-toxicity, cost efficiency, high stability in hydrogen plasma, and shallow donor levels due to the substitution of Zn2+ with Al3+(4,5). The objective of the study is to achieve precise control over the optoelectronic properties of AZO thin films through N doping. A facile sol-gel dip coating method has been employed for the fabrication of AZO thin films. Subsequently, post-thermal nitrification of AZO films will be examined by thermally decomposing NH3 at elevated temperatures to further modulate optoelectronic properties with N doping, which is considered the most promising p-type dopant with its similar electronic structure to oxygen and possessing a low ionization energy (6).

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*Speaker

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Keywords: TCO glass, AZO films, sol, gel

Spinel Glass Nanofibers for Application in Solid-State Batteries

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Lithium-ion batteries (LIBs) are a prominent industry, primarily due to their wide use, especially in electric vehicles (EVs). With the increasing expansion of EVs, demand is growing for recycling and reusing materials, especially valuable metals like lithium, nickel, manganese and cobalt. The demand for these metals is rising and spent LIBs can potentially serve as a secondary source. The predominant cathode materials used in LIBs for EVs are part of a category of layered mixed transition metal oxide compounds. These compounds are represented by the formula LiNixMnyCo1-x-yO2 (NMC).
NMC spinel appears as a promising material for use in solid-state batteries, representing a safer alternative to commonly available batteries with liquid electrolytes. The formation of glass nanofibers enhances safety and improves mechanical properties. The nanofiber structure is advantageous due to its flexibility and resilience, crucial during charging and discharging processes. NMC nanofibers were prepared using cathode material from commercial batteries from EVs; this reuse contributes to material recycling.

The LIB pouch cell was disassembled into individual layers of cathodes, anodes and separators. The cathode material including aluminium foil was manually cut into square pieces with a size of 25 × 25 mm. These layers were then placed in a porcelain crucible and heat treated in an electric furnace at a temperature of 400 ◦C for 60 min with a temperature rise of 5 ◦C/min in air atmosphere. Following thermal exposure and spontaneous cooling to laboratory temperature, the layers were placed in a container with distilled water, and using an ultrasonic probe, the active material was separated from the aluminium foil, serving as the current collector, on which the material is pressed. After drying, the material was prepared for nanofiber production. Electrospinning was employed, creating fibres using electrical voltage from polymeric precursors. For this experiment, a solution was prepared from tetraethyl orthosilicate (TEOS) and polyvinylpyrrolidone (PVP), enriched with NMC particles. The viscosity of the prepared solutions was measured, and the flow rate of polymeric precursors with particles during the spinning process was examined.

The final step in nanofiber preparation is high-temperature calcination. The individual fibre layers were pressed, and the electrochemical properties were measured. The resulting fibres were characterized using various methods. X-ray diffraction (XRD) and X-ray fluorescence (XRF) were used to verify composition. Fibre thickness was measured on a laser confocal microscope and scanning electron microscope, which also allowed to measure the thickness of individual layers.
This work was supported by the project "The Energy Conversion and Storage", funded as project No. CZ.02.01.01/00/22_008/0004617 by Programme Johannes Amos Comenius, call Excellent Research.

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Keywords: glass, ceramic, seal, hydrogen production, electrolysis, porosity, crystallization

Structural Characterization of Novel Tin Fluorophosphate Nuclear Salt Waste Forms

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Recently, phosphate glass, specifically tin fluorophosphate glass (TFPG), or SnF2-SnO-P2O5, has gained attention as a potential vitrification medium for chloride-based nuclear salt waste. Greater chemical durability and density along with lower melting temperatures imparts TFPG distinct advantages relative to borosilicate glass. These include better stability, decreased waste volume, and easier synthesis, which results in subsequent cost reductions for storage as well as fabrication. In this work, the inclusion of crystalline phases into TFPG was discovered to create glass-ceramic variants (TFPGC) that further stabilize their structural matrices against damaging environmental effects (i.e., hygroscopicity). Structural, topological, and composition analyses of the TFPG/TFPGC sample suites included XRD and SEM-EDS and contribute to our ongoing work in the development of waste immobilization materials.

Keywords: nuclear waste vitrification, tin fluorophosphate glass, tin fluorophosphate glass ceramic, chloride, crystalline phase, inclusion, structural characterization, XRD, SEM, EDS

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Study of a glass-ceramic seal : porosity and crystallization

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Study of a glass-ceramic seal : porosity and crystallization

Sealing is a major issue in High Temperature Electrolyzers (HTE) used for hydrogen production. The specifications that seals must meet are particularly demanding (thermal and chemical resistance, thermal expansion coefficient, electrical resistivity, viscosity...). These numerous constraints lead to the use of glass powder suspended in organic solvents, which is heat-treated to form a glass-ceramic seal.

Objectives of the study are to characterize and model the microstructure (porosity and crystallization) of the glass-ceramic seal. Indeed, a large and interconnected porosity can decrease the sealing properties. Moreover, a dense material will, a priori, be mechanically more resistant than a porous one. Finally, the crystallization may have a significant effect on the mechanical properties (resistance, tenacity, viscosity, hardness...).

The evolution of the microstructure as a function of temperature and duration of the heat treatment has been determined. Different crystals have been identified by X-Ray Diffraction (XRD) and electron microprobe. The crystalline surface fraction of the main phase has been determined by image analysis of SEM pictures using BackScattered Electron (SEM-BSE) detector. Its evolution has been modeled using a JMAK (Johnson-Mehl-Avrami-Kolmogorov) model which allowed to determine the crystallization kinetics. The evolution of porosity (percentage, mean diameter, number of pores) has been determined by image analysis of Scanning Electron Microscopy pictures using Secondary Electron detector (SE-SEM). This paper will present the obtained results.

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Keywords: glass, ceramic, seal, hydrogen production, electrolysis, porosity, crystallization

The second life of waste glass: Glass Microspheres production and versatile applications

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Glass recycling is far less straightforward than it appears. Using a cullet as a feedstock to fabricate original articles by remelting cannot always be applied. Some glasses are 'unrecyclable' for several reasons, including the risks of degradation of their properties or the loss of chemical purity. The main obstacle to reusing glass waste is that it should pass through various expensive and time-consuming steps. Technological progress is necessary to improve production efficiency, environmental quality, and economic competitiveness. Moreover, the development of new applications is required due to the non-biodegradable nature and increasing quantities of waste. Achieving certain goals can be accomplished by producing glass microspheres of various forms (solid, hollow, and porous) through innovative flame synthesis techniques for diverse applications.

Pharmaceutical glasses in a borosilicate system were used to fabricate novel transparent and porous 3D glass structures. Fiberglass waste was subjected to alkali activation in an aqueous solution with different sodium/potassium hydroxide concentrations. The activated materials were fed into a methane–oxygen flame with a temperature of around 1600 ◦C to produce porous glass microspheres. The highest homogeneity and yield of porous glass microspheres (PGMs) corresponded to the activation with 9 M KOH aqueous solution. The obtained PGMs were used to prepare highly porous pellets with a specific surface area of nearly 20 m2/g, which is applicable for removing methylene blue as a model organic dye from wastewater. Moreover, 3D photocatalysts containing PGMs doped with TiO2 nanoparticles have been fabricated using the masked stereolithography technique.

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Keywords: glass waste, recycling/upcycling, glass microspheres, additive manufacturing

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Industrial issues and solutions & Glass science to support sustainable development

Monday April 29

8:00-8:45	*Registration*	
8:45-9:00	*ICG SpringSchool introduction by organizing committee*	
	Industrial issues roadmap to reduce CO2 emission	
9:00-9:30	**Introduction by glass for Europe**	**Bertrand Caze**
9:30-10:00	**What have the Romans ever done for us? Two thousand years of glass recycling**	**Nadine Schibille**
10:00-10:20	**Decarbonized energy roadmap - Moving away from fossil fuels**	**Emre Dumankaya, Canalp Kulahlı**
10:20-10:50	*Coffee break*	
10:50-11:30	**Toward decarbonized specialty glass**	**Allison Yake**
11:30-12:10	**Decarbonization Roadmap applied to the Glass Fiber Industry**	**Anne Berthereau, Eric Dallies**
12:10-12:50	**Technical glass opportunities for a sustainable future and collaboration with academia**	**Jeff Kohli**
13:00-14:30	*Buffet at the hotel*	
	What is a Glass?	
14:30-15:10	**Basic data on silicate and borosilicate glass structure**	**Laurent Cormier**
15:10-15:50	**Basic data on glass structure and its influence on viscosity**	**Daniel Neuville**
15:50-16:30	**Glass and glass-ceramic formulation: an introduction**	**John Mc Cloy**
16:30-17:00	*Coffee break*	
17:00-17:40	**From melt to glass fibers**	**Yuanzheng Yue**
17:40-20:00	Teasing poster (3 minutes maximum for presentation, students only)	
20:00-22:00	*Buffet at the hotel*	

Tuesday April 30

	What are the current furnace technologies and technical solutions to reduce CO2?	
8:30-9:10	**The usefulness of modelling for improving energy efficiency**	**Malte Sander**
9:10-9:50	**Electrical melters : principle, design and limitations**	**Ankith Santosh**
9:50-10:30	**The decarbonation pathway of a borosilicate glass furnace: recorded successes and future challenges**	**Johann Brunie**
10:30-11:00	*Coffee break*	
11:00-11:40	**Current technologies nuclear waste vitrification furnaces/Technology and issues**	**Emilien Sauvage**
11:40-12:10	Open discussion	
	How to optimize the glass and glass-ceramic process?	
12:10-12:50	**Glass synthesis: Focus on Industrial and nuclear glasses**	**Sophie Papin and Richard Pokorny**
13:00-14:30	*Buffet at the hotel*	
14:30-15:10	**Structural design of borosilicate-based nuclear waste glasses**	**Ashutosh Goel**
15:10-15:50	**How the redox play for structure and properties: Implication for glass industry**	**Daniel Neuville**
15:50-16:30	**The role of chemical diffusion in glass processes and related challenges for glass sustainability**	**Emmanuelle Gouillart**
16:30-17:00	*Coffee break*	
17:00-17:40	**Modern computational methodologies for new Glass development**	**Alfonso Pedone**
17:40-18:20	**Accelerating glass discovery with artificial intelligence and machine learning**	**Anoop Krishnan**
20:00-22:30	Poster session #1 & Cocktail dinner	

Wednesday May 01

	What role does glass play in the energy transition? & a focus on mechanical properties	
9:00-9:40	**Glass for batteries**	**Virginie Viallet**
9:40-10:20	**Self-healing high temperature functional glass for hydrogen fuel cell sealing**	**Francois Mear**
10:20-11:00	**Challenge and progress in solar mirrors and glass for greenhouses**	**Mohammad Shayesteh**
11:00-11:30	*Coffee Break*	
11:30-12:10	**Observing mechanical and elastic properties of glasses by spectroscopy methods: toward weight-**	**Dominique de Ligny**
12:10-13:00	**Open discussion**	
13:00-14:00	*Buffet at the hotel - Free afternoon*	
20:00-22:00	*Buffet at the hotel*	

Thursday May 02

	What are the Primary raw material issues and the LCA methodology?	
8:30-9:10	**Responsible and secure supply of raw materials**	**Paul Notom**
9:10-9:50	**Life Cycle Analysis approaches developed for glass sustainability**	**Anna Maria Ferrari**
	What are the technical solutions to reduce CO2 emission?	
9:50-10:40	**Sustainable raw materials for glass production**	**Ateş Gösterişlioğlu,**
10:40-11:10	*Coffee break*	
11:10-11:50	**Sustainability in glass manufacturing: Contribution from silica and silicates**	**Hans Van Limpt**
11:50-12:30	**Schott Climate Neutral 2030 – A way to decarbonization in the glass industry**	**Wolfgang Schmidbauer**
12:30-13:10	**Various ideas and technical foundations for achieving carbon neutral glass melting**	**Maehara Terutaka**
13:10-14:30	*Buffet at the hotel*	
14:30-16:30	Poster session # 2	
16:00-16:30	*Coffee break*	
	How to optimize the energy efficiency and the foaming?	
16:30-17:10	**The usefulness of modeling for improving energy efficiency**	**Miroslav Polak**
17:10-17:50	**The contribution of thermodynamics in determining the parameters of elaboration and energy**	**Corinne Claireaux**
17:50-18:30	**Refining techniques and developments**	**Franck Pigeonneau**
20:30-23:00	Banquet - Cocktail dinner - Best student poster prize	

Friday May 03

9:00-9:20	**Improving container glass collection & recycling: a European perspectives** *(recorded presentation)*	**Vanessa Chesnot**
9:20-11:00	**Draft a roadmap: Glass for a sustainable future: How can glass scientists help meet the challenge?**	
11:00-11:30	**Conclusion & 11:30-12:30 Brunch**	

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Glass for a sustainable future

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