Session III, November 20, Wednesday

L9

STUDY OF LOCAL ATOMIC STRUCTURE OF DISORDERED MATERIALS USING *IN-SITU* SYNCHROTRON RADIATION

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The atomistic mechanisms behind the aging and rejuvenation of bulk metallic glasses (BMGs) are still unclear. Early studies on glassy polymers in the 1950s [1] and debates in the late 1990s [2,3] highlighted the challenges of aging, which increases brittleness and limits BMG applications. Various strategies, including deformation, high-pressure torsion, ion irradiation, flash annealing, and cryogenic cooling, have been explored to control aging and rejuvenation, improving mechanical properties. However, the fundamental atomistic origins of these effects remain unresolved.

Recent advances in computer simulations have shed light on the structural and thermodynamic origins of aging and rejuvenation in metallic glasses [4,5]. However, the kinetic aspects and experimental validation of these findings, especially those linking dynamic relaxation modes to atomic-scale reorganizations, remain limited. Different studies have explored correlations between stress-driven processes like shear transformation zones and dynamic relaxations, specifically - and -relaxation modes. A newly identified - or ’-relaxation mode, active at low temperatures, may be linked to stress inhomogeneities at cryogenic temperatures, but its structural origin is still unclear due to experimental challenges.

This contribution provides a deeper understanding of the relationship between structural reorganization and dynamic relaxations in glassy materials, particularly bulk metallic glasses (BMGs). Understanding and experimentally validating the atomistic mechanisms during aging and rejuvenation are essential for improving and explaining the limited ductility of BMGs. However, many structural characterization methods struggle to detect the subtle changes associated with these processes. In this study, we use in situ synchrotron X-ray diffraction to observe structural rearrangements during annealing, from 77 K to the crystallization temperature of $Cu_{44}Zr_{44}Al_8Hf_2Co_2$ BMGs. We introduce a method to visualize subtle changes in topological ordering by using a configurational entropy equivalent of the experimentally determined X-ray pair distribution function (PDF). The samples were rejuvenated through high-pressure torsion (HPT) at cryogenic and room temperatures prior to annealing. Structural changes, as indicated by the X-ray-derived equivalent configurational entropy, are correlated with dynamic mechanical analysis (DMA) and differential scanning calorimetry (DSC) to assess dynamic relaxations and crystallization. DMA measurements offer a detailed view of the relaxation processes, distinguishing between the well-known - and -relaxation modes and identifying the presence of the faster -relaxation mechanism in the glassy material.

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L10

PYROLYSIS-INDUCED STRUCTURAL CHANGES IN OLEIC ACID-COATED MAGHEMITE NANOPARTICLES

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Iron oxide nanoparticles (IONPs) are a class of materials with versatile applications, ranging from biomedicine [1] to catalysis [2] and environmental remediation [3]. To exploit their unique properties, such as high surface area, magnetic behaviour, and colloidal stability, IONPs are often synthesized and stabilized using oleic acid (OA) to achieve monodispersed size distributions [4,5]. The OA coating plays a crucial role in stabilizing the nanoparticles, preventing agglomeration, and enabling self-organization into mesocrystals with long-range order [6,7]. With their high surface area and atomistic alignment, mesocrystals are promising candidates for electrode materials in catalysis or battery applications. However, to enhance the surface activity of these materials, the removal or decomposition of the organic surfactant layer is necessary. Studying the temperature-dependent structural evolution of these oleic acid-coated IONPs is essential for understanding their thermal stability, phase transitions, and magnetic behaviour, thereby ensuring their practical utility.

In this contribution, we present our work on the evolution of magnetic phases in maghemite (-Fe_2O_3) nanoparticles upon the thermal decomposition of the surrounding oleic acid ligand layer in the temperature range between 20 and 1000 °C. Using *in-situ* synchrotron X-ray diffraction and total scattering, we observe a significant effect of varying amounts of oleic acid on the structural phase transitions in the iron oxide nanoparticles, transitioning from a spinel to rock salt to cubic iron. Our findings provide valuable insights into the thermal stability mechanisms of oleic acid-stabilized iron oxide nanoparticles, offering guidance for their effective utilization in diverse applications that require thermal robustness.

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We acknowledge DESY (Hamburg, Germany), a member of the Helmholtz Association HGF, for the provision of experimental facilities. Parts of this research were carried out at PETRA III, and we would like to thank Dr. Timo Müller for assistance in using the P21.1 beam line.

In the program, this lecture was replaced by contribution by Dominika Zákutná

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SAXS STUDIES OF MORPHOLOGY AND MICROSTRUCTURE OF THREE-DIMENSIONALLY ORDERED MICROMESOPOROUS CARBON

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The three-dimensionally ordered micro-mesoporous (3DOmm) carbon with ordered spherical mesopores and micropores in the walls was synthesised and activated by treating it at high temperatures between 973-1173 K under CO_2 flow. The evolution of morphology and microstructure of 3DOmm material was studied as a function of activation temperature using the SAXS. A relevant physical model was developed for the 3DOmm material: we assumed the spherical clusters of 3DOmm carbon having a mean size *Rc* and root mean square deviation of clusters radii *Rc*. The clusters are filled with an ensemble of spherical bubbles arranged in face centered cubic (fcc) lattice with centers of bubbles displaced by distance *D* and root mean square deviation *D*. Individual bubbles having an internal core-shell structure with diameter *R*, root mean square deviation *R* and shell thickness *d*. The SAXS intensity is assumed as a weighted sum of intensities calculated using the decoupling (DA) and local monodisperse (LMA) approximations. In studied samples the mean cluster radius *Rc* lies around 280 nm. The mean distance of the bubbles centers *D* is around 28 nm, which represents the lattice parameter of the fcc structure, around 40 nm. The mean bubble radius decreases from 9 nm in 3DOmm sample to about 4.8 nm in sample 3DOmm-1173 K.

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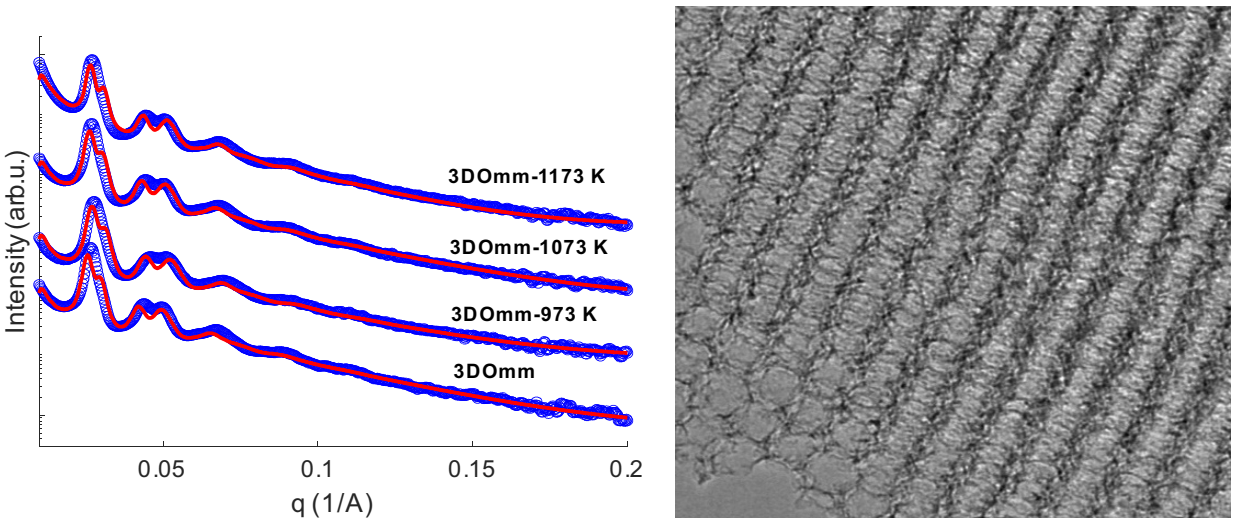

Figure. 1. Measured (open blue circles) and fitted (red lines) SAXS profiles of the 3DOmm carbon materials (a). HRTEM micrographs of the 3DOmm sample (b).