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**Organizing Contact Point**

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# In Situ TEM to quantify thermal activation parameters of plasticity

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**Résumé**

In situ Transmission Electron Microscopy allows to both change stress and temperature and follow simultaneously the velocity of dislocations. Thermal activation theory, that links the stress, temperature, and strain rate can serve to get access to activation volume and activation energy directly using these observations

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# Continuous in situ high-resolution digital image correlation (DIC) during microtensile testing

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**Résumé**

Digital Image Correlation (DIC) is widely used across length scales to map surface strain fields on deformed materials. At the micro- and nanoscale, high-resolution DIC (HR-DIC) can resolve localized strains associated with discrete plastic events such as crystallographic slip or interface debonding. Conventionally, HR-DIC studies in the scanning electron microscope (SEM) are performed in an interrupted (quasi in situ) manner on millimeter-scale polycrystalline specimens, because stable high-magnification imaging of large areas during continuous loading is challenging.

This contribution presents a methodology for truly continuous HR-DIC during quasi-static microtensile deformation in the SEM. Continuous strain mapping is achieved through three advances: (i) optimized electron-beam-induced deposition of high-contrast platinum nanoscale speckle patterns suitable for short exposure times and fast frame rates, (ii) focused ion beam (FIB) fabrication of micrometer-scale freestanding tensile specimens that keep the region of interest within the SEM field of view throughout the test, and (iii) automated SEM remote-control software that enable continuous high-magnification image acquisition during quasi-static deformation.

The approach is demonstrated on single-crystal and multilayer thin-film microtensile specimens, revealing real-time evolution of global elastic strains and of plastic strain localization phenomena that are inaccessible with interrupted testing protocols. The method significantly improves temporal resolution of deformation and opens new possibilities for quantitative in situ nanomechanical studies of small-scale materials.

**Mots-Clés:** DIC, tensile testing, in situ testing

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# Contribution of slip, twinning and de-twinning to the mechanical deformation of Haynes 244 alloy at 23ºC and 650ºC

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**Abstract**

Haynes 244 is a Ni-based superalloy that contains 40 vol.% of coherent Ni2(Cr, Mo, W) precipitates with a lenticular shape. The deformation mechanisms of this alloy were investigated at 23ºC and 650ºC by means of micropillar compression tests in grains with different orientations, suitably oriented for dislocation slip or twinning. The mechanical response of the grains oriented for slip at room temperature showed strain bursts, strain hardening, and a fully elastic recovery upon unloading, which are indicative of dislocation plasticity. On the contrary, micropillars oriented for twinning did not show strain bursts or strain hardening and presented a large strain recovery upon unloading. In situ mechanical tests provided evidence of twin nucleation and thickening during compression, followed by detwinning during unloading. Micropillar compression tests in both orientations at 650ºC showed similar features: strain bursts during deformation, limited strain hardening, and elastic recovery during unloading. The deformation mechanisms of the micropillars deformed in both orientations and at both temperatures were carefully analyzed by transmission electron microscopy from lamellae extracted from the micropillars. In particular, the origin of the detwinning found upon unloading at ambient temperature was discussed in terms of the microstructure of the alloy.

**Keywords:** Ni alloy, Micropillar compression, Detwinning, Deformation mechanisms

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# Cyclic deformation mechanisms associated with planar faults and dislocation decorrelation in Alloy C263

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**Résumé**

Understanding the evolution of planar defects is essential for linking deformation mechanisms to fatigue response in nickel-based superalloys. In Alloy C263, the occurrence and role of stacking faults (SFs) remain unresolved. Observations conducted by transmission electron microscopy in this work reveal that SFs are already present in the post-heat-treated state and can still be identified even after the isothermal testing at 800∘C. This finding contrasts with the current understanding that SFs in C263 appear only after high-temperature isothermal or creep testing between 450 and 900 ∘C. However, this work demonstrates that, with longer exposure at 800 ∘C in a lower strain-amplitude regime, the stacking faults progressively diminish through dislocation decorrelation and segment annihilation. This behaviour during isothermal testing can be explained by diffusion-assisted decorrelation of the two $a/6\langle 112\rangle$ partials bounding the intrinsic stacking faults. At elevated temperature, diffusion-driven climb and solute segregation modify the local stacking-fault energy and effective stress on each partial, so that they no longer move as a coupled pair. Differences in their effective driving forces, arising from the Orowan stress, the local stacking-fault energy and the $\gamma$-channel width, lead to a decorrelation of their motion, producing a widened and eventually fragmented fault ribbon in the $\gamma$ matrix. In contrast during thermomechanical fatigue (TMF, 300–800 ∘C) these defect practically vanished from microstructure and twins emerged. Moreover serrations were observed in the hysteresis loops, indicating a possible link between the cyclic stress–strain response and microstructural development. It is presumed that at elevated temperature, matrix dislocations can cross-slip onto existing fault planes, where their repeated interactions generate extrinsic or superlattice extrinsic stacking faults ((S)ESFs). These SESFs act as precursors to twin formation. The twins then develop through the glide of successive partial dislocations on adjacent {111} planes within the faulted regions. Each advancing partial reorders the atomic planes, converting the faulted structure into a coherent twin. This sequence is further facilitated by segregation of Cr and Co atoms, which lowers the local stacking-fault energy and stabilizes faulted structures, allowing the twin to thicken

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progressively. Segregation of Cr and Co not only assists fault thickening but also causes dynamic strain ageing through the pinning and unpinning of partial dislocations by solute atmospheres, producing the typical serrated mechanical response. To directly verify these mechanisms, in situ TEM tensile tests at room temperature, 500 ◦C, and 800 ◦C will be requested using a quantitative straining holder to synchronize mechanical data (serrations) with high-speed defect visualization. Furthermore, quantitative stress relaxation tests at 500 ◦C and 800 ◦C will determine the activation volume, providing the key kinetic signature to link the serrated flow to the diffusion and pinning mechanisms of Cr/Co solute atmospheres.

**Mots-Clés:** Nickel, based superalloy, stacking faults, twinning, high, temperature fatigue, dislocation decorrelation, dynamic strain aging

# In situ SEM nanomechanical testing of nanowires using a versatile robotic platform

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**Résumé**

A variety of nanomechanical tests are realised using a custom-built robotic platform installed inside a focused ion beam - scanning electron microscope (FIB-SEM) system, which provides a level of versatility that goes beyond that offered by commercial *in situ* SEM nanoindentation instruments. Three distinct in situ SEM configurations with increasing technical complexity are presented. First, a nanowire (NW) peeling test is realised for characterising the adhesion of one-dimensional interfaces. Here, quantifying the work of adhesion relies entirely on SEM observation of a NW's deflection shape as it detaches from a substrate. Second, uniaxial NW tensile tests and nanobeam-scaffold compression tests are realised to characterise fracture strength and to identify the point of failure within a scaffold, respectively. Force-displacement curves are constructed from a capacitive force sensor and stage positioning sensors, which can be used in parallel with SEM imaging. To induce tension in a NW, glueing of the NW directly to the force-sensor tip using electron beam induced deposition (EBiD) is required. Finally, high-throughput NW tensile tests are realised through the integration of NWs into custom-designed silicon-on-insulator (SOI)-based microelectromechanical systems (MEMS), which are subsequently actuated via a capacitive force sensor under SEM observation. This approach facilitates the testing of multiple NWs within a MEMS array, with reduced alignment steps and without the need of EBiD glueing directly to the force sensor. Presentation of these three distinct techniques illustrates the variety of nanomechanical tests that can be realised inside an SEM and also highlights how the application of MEMS towards nanomechanical testing can simplify workflows and increase experimental throughput.

**Mots-Clés:** nanowire, in situ SEM, nanomanipulation, nanomechanics, fracture strength, adhesion energy

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# Micromechanical integrity of High Entropy Carbide based cemented carbides

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**Résumé**

High Entropy Alloys (HEA), mixture of an equal or relatively large portion of five or more elements are presented as a good replacement providing good wettability, high toughness, wear resistance, and temperature stability. Inspired by the concept of HEA, high entropy carbides (HEC) have gained attention in recent years; however, their mechanical integrity in cemented carbides remains poorly understood.

In a previous study, detailed insights into phase mechanical properties, phase volume quantification and their microstructural representation from each individual mechanical phase, (carbide and binder) was analysed by mean of high-speed nanoindentation mapping technique. Nevertheless, the integrity of the carbide-carbide and carbide–binder interfaces, which represent critical factors in the performance of these composite materials, was not characterised.

In the present work, the microstructure and micromechanical properties of a (HEC) composed of (Ti, Ta, Nb, V, W) combined with two different binders (Ni and Co) were investigated. Particular attention was given to interfaces. Hence, micropillars were fabricated by FIB milling in both cemented carbide grades and subsequently in-situ tested in a Femtools nanoindenter NMT04. Fractographic inspection of the compressed pillars was correlated with the corresponding load–displacement curves. Furthermore, by integrating the results from the previous study-hardness (H), elastic modulus (E), and H/E ratio maps obtained through high-speed nanoindentation mapping and statistical analysis-the micromechanical integrity of the HEC cemented carbides is comprehensively described.

**Mots-Clés:** High Entropy Carbide, Micropillars, Compression, High speed nanoindentation

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# Understanding nanoindentation statistical dispersion in ceramic–metal cemented carbides by numerical simulation and FIB tomography

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**Résumé**

Understanding the mechanical response of biphasic materials requires not only characterizing the individual behavior of each phase but also their interaction within complex microstructures. When phases exhibit similar mechanical properties or intricate spatial distributions, statistically separating them in indentation maps becomes challenging, complicating data interpretation. In this study, the WC–Co system is used as a representative case. Simulated nanoindentations-generated from three-dimensional reconstructions obtained by FIB/SEM tomography-are combined with experimental hardness maps, SEM imaging, and real cross-sectional cuts of indentations prepared using FIB/SEM. A total of 128 realistic 3D meshes were simulated at various indentation depths and directly compared with experimental results. The findings show a strong correlation between hardness and the local 3D binder content around the indentation, whereas 2D binder fractions exhibit substantially higher scatter. Moreover, integrating simulations with FIB/SEM cross-sections enables direct assessment of how hardness evolves with the local microstructure near the indentation, providing a more accurate interpretation of the material's mechanical response.

**Mots-Clés:** Nanoindentation, Composite material, WC, Co, Binder, FIB/SEM, 3D Tomography, FEM Simulation, Microstructure

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# Integrating Sensor-Driven Signal Modeling and Machine Learning for Enhanced Interpretation of In Situ Electron Microscopy Experiments

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**Résumé**

In situ electron microscopy enables unprecedented observation of the deformation behavior of crystalline and metallic materials under controlled loading. However, the complex and high-dimensional data streams produced during nanoindentation, microcompression, and TEM-based traction tests present challenges in quantitative interpretation and reproducibility. This presentation proposes a complementary framework that integrates sensor-based signal modeling, wireless data acquisition principles, and machine-learning-driven feature extraction-methods widely used in IoT, structural monitoring, and high-frequency signal analysis. Building on recent work involving LIDAR-derived spatial mapping, noise-aware signal loss modeling, and machine-learning-supported deformation parameter estimation, the study introduces transferable analytical tools that can enhance the understanding of stress–strain evolution, defect dynamics, and material response observed during in situ microscopy. The talk will discuss how advanced denoising, spatio-temporal pattern recognition, and automated event detection pipelines can support quantitative interpretation of SEM/TEM experiments, reduce operator bias, and enable hybrid datasets that merge microscopy outputs with external sensor models. This cross-disciplinary perspective aims to contribute to more scalable, data-rich, and reproducible use of in situ electron microscopy in the characterization of structural materials.

**Mots-Clés:** spatio, temporal modeling, machine learning, signal processing, structural materials, defect dynamics, IoT, inspired data acquisition, pattern recognition

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# UNDERSTANDING LATENT HARDENING MECHANISMS IN FERRITE VIA CORRELATIVE CONTROLLED ELECTRON CHANNELING CONTRAST IMAGING AND MICRO-PILLAR COMPRESSION

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**Résumé**

Forming processes used to shape materials typically involve multiaxial loading and changes in the load path. Mechanistically, such variations activate different slip systems, and mutual interactions between previously and newly activated slip systems give rise to latent hardening, i.e., a form of hardening governed by slip system interactions. A comprehensive understanding of this mechanism is required to accurately model material behavior and design damage-tolerant steels. In dual-phase steels such as DP800, previous studies have linked the strain hardening capacity of the ferrite phase to the suppression of void growth, assuming isotropic hardening (1). However, this assumption overlooks the path-dependent nature of latent hardening, limiting the prediction of the material behavior under complex loading conditions.

In this study, we present the latent strain hardening capability of ferrite studied by *in situ* scanning electron microscope (SEM) micro-pillar compression tests and controlled electron channeling contrast imaging (cECCI). To understand the latent hardening of ferrite, firstly, an oligocrystal was macroscopically pre-strained in tension. Lüders Band formation was observed in a section of the gauge length; therefore, the subsequent analysis was done inside the Lüders Band, where the deformation is localized. Local dislocation characteristics, including Burgers vector and local dislocation density, were analyzed using cECCI and machine-learning-based instance segmentation in a high-throughput manner. Subsequently, it was directly correlated with the local critical resolved shear stress (CRSS) measured by micro-pillar compression. An increase in the CRSS after pre-straining, following Taylor hardening fitting, was observed. Additionally, an activation of a secondary slip system was visible in the last stage of deformation (approximately 8% of strain), and after analysis of the post-mortem images, it was found that the activated slip system shares the same direction as the dislocations activated after pre-straining, presuming cross-slip. A mechanism capturing

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the two stages of the observed latent hardening is proposed to serve as a micromechanical framework for modeling activities.,

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**Mots-Clés:** Latent hardening, micro, pillar compression, cECCI

# Toughening mechanisms and fracture behaviour in metallic–oxide nanolaminates revealed by in situ TEM tensile testing

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**Résumé**

In-situ tensile testing within a transmission electron microscope (TEM) offers unparalleled real-time insight into atomic-scale deformation processes, enabling direct correlation between mechanical response and microstructural evolution (1). In this work, we use this approach to elucidate toughening mechanisms and interface-driven behaviour in two classes of nanolaminates. For Cu(Al) multilayers incorporating ultrathin amorphous AlO interlayers, tensile experiments reveal how these ceramic layers enhance system-level toughness (2). Multilayers composed of 30–120 nm Cu–10%Al layers separated by 2 nm Al2O3 deposited via atomic layer deposition exhibit preferential void nucleation at interfaces between the ALD layer and high-angle grain boundaries, promoting tortuous crack trajectories. Ahead of the crack tip, multiple deformation processes operate simultaneously, including dislocation glide, grain boundary sliding, detwinning, and grain rotation. Remarkably, the nominally brittle Al2O3 interlayers display unexpected ductility when confined between metallic layers, thinning from 2 nm to roughly 500 pm prior to failure.
A complementary study of amorphous SiO2/Ta2O5 nanolaminates with bilayer thicknesses ranging from 2 to 200 nm highlights the influence of interface quality on failure behaviour. This includes a unique 2 $\mu$m-thick specimen consisting of 1000 exceptionally smooth 2 nm-thick laminates. Below a 20 nm bilayer periodicity, an inverse Hall–Petch trend emerges, with strength decreasing as the number of interfaces increases. The high-quality interfaces also enable clear visualization of shear band propagation and its coupling to mechanical response. Together, these results advance the understanding of how nanoscale architectural control and interface engineering can be leveraged to design damage-tolerant multilayer materials, spanning metallic–ceramic systems and fully amorphous oxide structures.

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**Mots-Clés:** In situ TEM tensile testing, nanolaminates

# The Ductility of Thin Freestanding Metallic Films investigated by in-situ TEM and in-situ AFM Nanomechanical Testing

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**Résumé**

Freestanding metallic thin films are generally thought to be very brittle and little is known about their ability to accommodate large strains under certain circumstances. New insights are provided by investigations on 30-200 nm thick PVD gold and silver films by means of in-situ tensile tests in a TEM and in-situ bulge tests in an AFM.

The bulge tests reveal that the low toughness of the films derives from the limited expansion of the fracture process zone, which limits the plastic energy dissipation. In-situ tensile testing in the TEM suggests that grain boundary mediated deformation mechanisms can significantly increase the tolerance of the samples to small cracks and lead to a ductility well above 10% plastic strain. These high ductility values measured on TEM samples are in sharp opposition to the behavior of larger samples, which typically fail around 1% strain. This is likely a statistical size effect, associated with the much larger surface of the bulge samples and the higher probability that they contain critical defects. As avoiding these defects is hardly achievable in practice, a promising strategy consists in improving the resistance of the films to crack nucleation and propagation. Using the bulge test technique, it was found that introducing grain boundaries and maximizing the material strength is key to optimizing the fracture toughness of freestanding metallic films.

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**Mots-Clés:** thin films, nanomechanical testing, TEM, AFM

# In-situ TEM investigations of visco-plastic mechanisms in nanocrystalline materials using (on-chip) micro-mechanical testing

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**Résumé**

The improvement of the thermomechanical performances of coatings is more than ever a major challenge in, among others, the energy production, transportation, environment and biomedical engineering fields which many components undergo severe tribological solicitations. Thin films also emerge as a solution to protect bulk materials against the environment. Over the last years, the on-chip tensile testing method developed at UCLouvain has proven its potential for the characterization of the link between the mechanical behaviour and microsctructure at the micro- and nanoscale. Using specific design, in-situ TEM observations have supplemented the analysis to understand the local deformation and relaxation mechanisms. An overview of the latest outputs obtained using the residual stress actuated on-chip method will be presented. A comparison of this method with other in-situ mechanical testing techniques will also be discussed.

**Mots-Clés:** thin film, mechanical properties, in, situ micromechanical testing

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# Stress-induced amorphization and grain-boundary sliding in olivine

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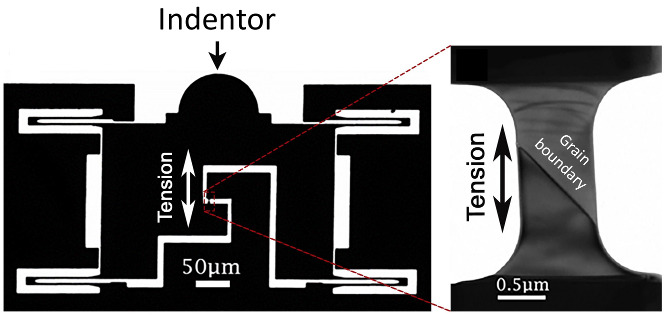
Stress-induced amorphization has recently attracted attention as a potential deformation mechanism in ceramics, semi-conductors or minerals [1]. Its activation is promoted when conventional plasticity, *e.g.* driven by dislocations, is inhibited, however the underlying mechanisms are still unclear. In this study, we focus on olivine, a magnesium-iron silicate that is a major constituent of the Earth's upper mantle. Using quantitative *in situ* TEM tensile testing, we demonstrate that stress-induced amorphization can be activated under high stresses at room temperature in small-sized olivine bi-crystals. High-angle grain boundaries are the preferred sites for amorphization whereas low-angle grain boundaries are less prone to this phenomenon. Amorphized grain boundaries are then subject to sliding and the resulting constitutive equation of such a sliding boundary has been determined. We show by Molecular Dynamics that this phenomenon can be considered as a case of transformation plasticity. Varying the iron content in olivine demonstrates that iron inhibits amorphization and, consequently, promotes brittle failure. This is in contrast to the accepted view at high temperatures where iron promotes ductility in olivine. These findings coming from natural minerals provide a novel approach to the control of the mechanical properties of grain boundaries in hard materials at low temperatures following a geomimetic inspiration.




**Keywords:** Amorphization, Grain boundary sliding, Transformation plasticity, Olivine

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# In-situ electron microscopy investigation of Cu thermo-mechanical fatigue

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**Abstract**

Managing metallization fatigue is essential to guarantee the robustness of power microelectronic devices. Since some failure modes are so severe that a precise failure analysis is no longer possible, it is indispensable to develop methodologies that allow studying the evolution of such degradation at early-stage. This work presents both an in-situ scanning electron microscopy (SEM) and an in-situ transmission electron microscopy (TEM) approach to study the thermo-mechanical behavior of a Cu metallization on a Si-based microelectronic test chip with integrated active heating. By applying short voltage pulses, heating rates up to 1 million kelvin per second can be achieved, replicating thermo-mechanical stress conditions that would typically occur during a short-circuit event in a power device. Utilizing the in-situ SEM setup at KAI GmbH, stress tests have been carried out, and Cu degradation has been tracked through automated SEM image acquisitions and electrical resistance measurements. In doing so, the effect of different testing parameters, e.g. heating rate, on the crack formation behavior was studied. To understand the involved physical processes leading to the thermo-mechanical fatigue of Cu to a fundamental level, in-situ TEM experiments have been performed at CEMES-CNRS. These experiments utilized a sample that was carefully prepared from the aforementioned microheater test chip so that its capability of electrical heating is retained. It is shown that upon heating, the thermo-mechanical stress built up by the system leads to movement of dislocations in the Cu governing deformation and degradation.

**Keywords:** Cu, thermo, mechanical fatigue, in, situ characterization, SEM, TEM

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# Electrical resistivity analysis of 3D additive manufactured copper pillars with different microstructures

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**Résumé**

3D micro-scale structures fabricated by bottom-up additive manufacturing methods possess novel mechanical and electrical properties that have yet to be discovered. While microstructures affect the resistivity of metal materials, this property remains unexplored in 3D-manufactured geometries. This study develops an in-situ method to analyse the relationship between the resistivity and microstructures of 3D materials. We use the electrochemical deposition method to create 3D copper pillars with different grain sizes, resulting in both micro- and nano-structure. The in-situ SEM testing incorporates a 4-probe measurement technique with a nanoindentation setup designed to analyse the electrical resistivity changes of the deformed 3D material. Preliminary results show the resistivity of the 3D copper pillar is comparable with the reference value, while further analysis can reveal the effect of microstructures and deformation on the electrical resistivity. These insights support the understanding of 3D metal properties in interconnects for electronic devices.

**Mots-Clés:** In situ SEM, electrical resistivity measurement, microstructures, additive manufacturing

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