PULSED LASER DEPOSITION RESEARCH CLUSTER FOR ENERGY MATERIALS

The pursuit of big science requires the realisation of increasingly complex and technological advanced systems. In industry, systems engineering has emerged as a discipline aimed at mitigating uncertainty and improving control of cost and time in large-scale, highly complex engineering projects. The Dutch Institute for Fundamental Energy Research (DIFFER) recognises the importance of systems engineering in developing complex systems and is therefore adapting the associated methods and tools for developing nuclear fusion power plants and their own research infrastructure. This article provides an overview of the insights gained in applying systems engineering tools and methods during the development of a pulsed laser deposition research cluster.

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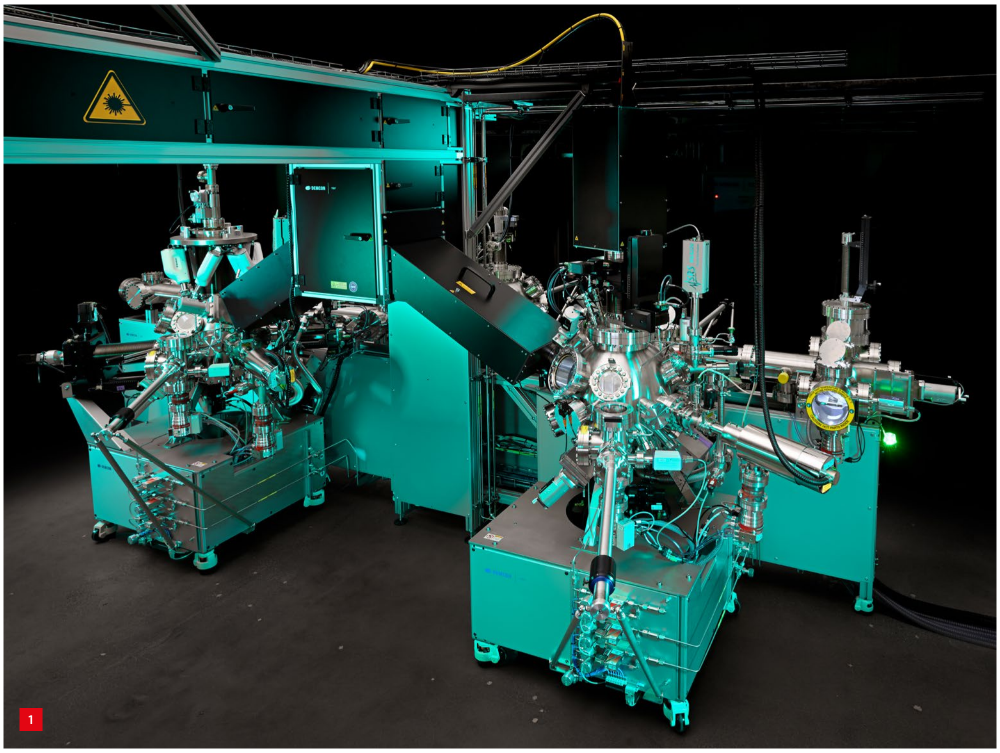
The pursuit of 'big science' frequently necessitates the development of state-of-the-art systems that challenge the limits of technology. Budgetary and scheduling overruns are frequent and substantial and these are commonly regarded as inevitable. In industry, however, systems engineering has emerged as a discipline aimed at mitigating uncertainty and improving control of cost and time in large-scale engineering projects.

The Dutch Institute for Fundamental Energy Research (DIFFER) recognises the importance of systems engineering and advocates the integration of systems engineering methodologies into big science initiatives. In collaboration with Eindhoven University of Technology (TU/e) and Ratio Computer Aided Systems Engineering (Ratio CASE), DIFFER is therefore adapting these methodologies for the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The pulsed laser deposition research cluster basis realised by Demcon TSST. (Photo: DIFFER / Bart van Overbeeke)

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development of nuclear fusion power plants as well as for designing and developing its own research infrastructure for both the Fusion Energy and Chemical Energy departments.

A recent milestone in this effort is the realisation of the basis of the pulsed laser deposition (PLD) research cluster for energy materials, shown in Figure 1. The system architecture, specifications, and conceptual design for the PLD cluster were created at DIFFER in close cooperation with the Inorganic Materials Science group of the Science and Technology Faculty of the University of Twente (UT). Subsequently, Demcon TSST created the detailed design and built the PLD research cluster basis, for which they had won the tendering procedure in January 2024. Twente Solid State Technology (TSST), founded in 1998 and part of the Demcon group since 2018, has decades of experience in designing and building PLD systems and has been a very valuable and cooperative partner.

The PLD cluster represents the first infrastructure at DIFFER to be developed using a systems engineering approach, encompassing all phases from conceptualisation and tendering to realisation, testing, and commissioning in cooperation with multiple contractors. In the forthcoming months, the research cluster basis will be extended with additional diagnostic capabilities to characterise thin films during the growth process.

PLD research cluster

The PLD research cluster was conceived as a user facility for the development and investigation of metal-oxide thin films derived from earth-abundant materials. These films are critical to the energy transition, offering potential improvements in cost efficiency, performance and durability across applications such as catalysis, photovoltaics, energy storage, energy conversion, optics, and microelectronics.

Prior to this initiative, the Netherlands lacked a research infrastructure capable of supporting the controlled upscaling of high-quality thin films. Specifically, no facility existed that enabled precise tuning and in-situ characterisation of composition, structure and opto-electrical properties in a reproducible and efficient manner on an industrially relevant scale. To address this gap, DIFFER and partners secured a €4.7 million grant from the Dutch Research Council (NWO) to develop the cluster (see acknowledgement in the Authors' note).

The facility comprises of two PLD systems, one for small-area substrates up to one inch in diameter and one for large-area substrates up to four inches, a sputtering system for the deposition of metal layers, and an X-ray photoelectron spectroscopy (XPS) system. These components are interconnected via a linear ultra-high-vacuum transfer line, facilitating contamination-free sample transport.

Pulsed laser deposition (PLD): principles and capabilities

PLD is a physical vapour deposition technique employed for the fabrication of thin films. A high-power pulsed laser beam – typically operating at a wavelength of 248 nm with a repetition rate of approximately 10 Hz and a pulse duration of 20 nanoseconds – is focused onto a target material within a vacuum chamber. The target undergoes rapid sublimation, and the ejected material forms a plasma plume that propagates towards a heated substrate, where it condenses to form a thin film (Figure 2).

The deposition process may occur under vacuum or in the presence of a reactive background gas, such as oxygen or nitrogen. Metal-oxide films produced via PLD typically exhibit thicknesses ranging from 5 nanometer to 1 micrometer. Depending on the deposition temperature (ranging from ambient to 1,000 °C) and the intrinsic properties of the material, the resulting films may be amorphous or crystalline.

Compared to alternative thin-film deposition techniques, PLD offers superior control over film microstructure and composition. Parameters, such as porosity, surface area, stress, strain and thickness, can be finely tuned. The technique also minimises interface damage and enables cold epitaxial growth, making it particularly suitable for multifunctional thin-film applications. Furthermore, the small target volume required facilitates efficient material screening, including the deposition of films derived from naturally occurring minerals.

Recent advancements have extended PLD's applicability beyond the laboratory, enabling commercial-scale fabrication of wafers up to 30 cm in diameter. PLD complements other deposition methods such as sputtering (optimised for metallic films), atomic layer deposition (ALD, ideal for ultra-thin conformal coatings), and plasma-enhanced chemical vapor deposition (PE-CVD, suited for thicker films).


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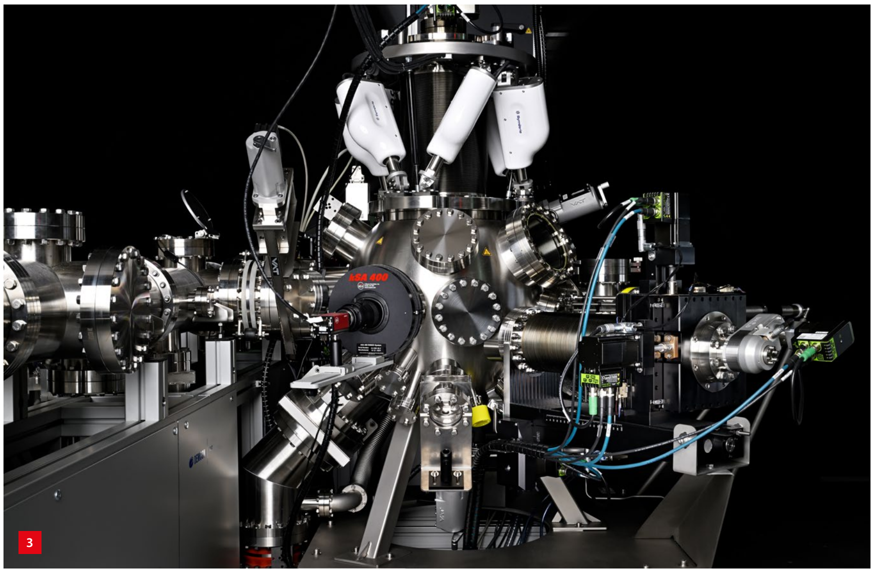
Plasma plume inside the small-area PLD chamber. (Photo: DIFFER / Bart van Overbeeke)

Both PLD chambers are equipped with a comprehensive suite of in-situ thin-film characterisation tools and auxiliary systems designed to monitor and control the deposition process and the resulting thin-film properties. These include reflective high-energy electron diffraction (RHEED) for assessing growth modes and crystallinity, spectroscopic ellipsometry (SE) for probing dielectric and optical properties, and an optical tensile sensor (OTS) for measuring substrate curvature for residual stress measurements.

A low-angle energy-dispersive X-ray spectroscopy (LA-EDS) module is currently under development in collaboration with Sioux Technologies to enable in-situ compositional analysis. Second harmonic generation (SHG) is being evaluated for its potential to characterise ferroelectric properties, while optical emission spectroscopy (OES) will be added to analyse the plasma during ablation.

In addition to diagnostic instrumentation, the cluster integrates a wide array of auxiliary systems, including vacuum and gas-handling equipment, substrate and target manipulators, heating elements, UV laser optics, sputter sources, power supplies, safety interlocks, loading and unloading mechanisms, substrate in-vacuum storage, and the requisite control hardware and software.

In total, the cluster comprises several hundred components, ranging from large commercially available modules, such as the XPS system, to semi-custom and fully bespoke designs. One example of the latter is a custom-designed hexapod substrate stage for heating and 6-degrees-of-freedom movement of four-inch substrates.



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The large-area PLD system with a custom hexapod stage for 6-degree-of-freedom in-vacuum motion of a 4" wafer. The wafer is positioned face down, 2.5 cm above the centre of the vacuum chamber. The stage was conceptualised at DIFFER, while Demcon TSST and Symétrie made the detailed design and built it. (Photo: DIFFER / Bart van Overbeeke)

The systems engineering process: top-down versus bottom-up design

Conventional systems engineering practices advocate a top-down design approach, beginning with the definition of high-level functional requirements. In our experience, however, this approach does not align well with the mindset of researchers who typically have a background in physics or chemistry. That is, they tend to have a bottom-up perspective. Their focus is typically directed towards the physical phenomena they aim to induce, observe and control, and the specific components required to facilitate the experiments.

From the researchers' standpoint, this approach is pragmatic. During the initial design phase, their primary objective is to secure funding through competitive proposals that emphasise scientific merit and provide reasonably detailed cost estimates. A comprehensive overarching system architecture and a conceptual design is not expected at this stage. Hence, creating such a design could result in a significant sunken cost if the funding is not granted.

Nevertheless, estimating the cost of a novel, potentially first-of-its-kind system is inherently challenging. Researchers therefore rely on known components – identified through prior experience or literature – that are suitable for their intended applications. These components are typically highly specialised, available from a limited number of suppliers, and function reliably only under specific conditions. Consequently, a bottom-up integration strategy often feels more intuitive and manageable.

However, the absence of an overarching system architecture and conceptual design in bottom-up approaches can lead to significant integration challenges. The reconciliation of these two paradigms – top-down and bottom-up – is essential in applying systems engineering to big science projects and reduce budget and schedule overruns.

For a systems engineer it is essential to dive into the details and quickly obtain a basic understanding of the physics of interest and the working principles of the various diagnostics. Otherwise, it is impossible to ask the right questions to figure out what the researchers want, what they truly need, what the critical performance parameters are, and how the various ideas can be integrated and addressed within an overall system architecture and conceptual design.

As such, as a systems engineer you have to constantly switch between a bottom-up perspective, in which you have detailed discussions on physics and working principles of individual components, and a top-down perspective in which you try to create an overall system architecture and conceptual design that meets all needs and requirements.

Listing 1

ESL goal-requirement specification example.

```
goal-requirement
 sut-provide-cooling-water-03: the_site_utilities shall provide cooling-water-flow-SAP to the_small_area_pld_system
 with subclauses
   * s1-medium: cooling-medium shall be equal to water [-] #< Filtrated, mechanically clean, optically clear,
     no turbidity, no sediments, chemically neutral.
   * s2-max-conductivity: maximum-flow-conductivity shall be equal to t.b.d. [S/m] # Optional
   * s3-max-inlet-temperature: maximum-inlet-temperature shall be equal to 25 [degrees_Celsius]
   * s4-min-inlet-temperature: minimum-inlet-temperature shall be equal to 15 [degrees_Celsius]
   * s5a-nominal-flow-rate: cooling-water-flow-SAP.MTP shall be equal to 100 [l/hour]
   * s5b-nominal-flow-rate: cooling-water-flow-SAP.RTP shall be equal to 75 [l/hour]
   * s5c-nominal-flow-rate: cooling-water-flow-SAP.LLTP shall be equal to 75 [l/hour]
   * s6-nominal-heat-load: nominal-heat-load shall be equal to t.b.d. [W]
   * s7-inlet-pressure: inlet-pressure shall be at most 6 [bar]
   * s8-max-pressure-drop: maximum-pressure-drop shall be equal to t.b.d. [bar]
   * s9-device-coupling-type: device-coupling-type shall be equal to 8_mm_plug-in_connection [-]
   * s10-hardness-type: water-hardness shall be at most 10 [degree_dH]
   * s11-pH-range: water-pH-range shall be equal to 7-9 [-]
```

This is an iterative process during which the researchers' needs and requirements may change depending on the feasibility and compatibility of the various design concepts. Moreover, it is important that alternative technologies and designs are actively explored and discussed with the researchers to avoid running into a design funnel too soon and to obtain a well-motivated overall design. Design decisions made for detailed components have implications for the full system and should be actively investigated and discussed with the scientists. A model-based approach herein is essential as is described in the next section.

The process is characterised by many short-cycled design changes. For example, in designing the vacuum vessels for the PLD chambers, as shown in Figure 3, over fifty different versions were created. In this stage, however, making design changes to resolve integration issues or to provide additional functionality is cheap as it is purely an on-paper exercise. Hence, it is worth the effort to take a prolonged period of time for this process before the detailed design phase starts.

For the PLD research cluster, this process took place in the period between the submission of the proposal in February 2022 and the official start of the project in October 2023, during which a three-person core team worked on it for on average two days a week, supported by several disciplinary experts for a few hours a week. The funding was officially granted in May 2023.

This pre-work provided the basis for a swift detailed design and realisation phase. It took only twenty months, including a five-month tendering procedure, from the official start of the project in October 2023 to delivery of the research cluster basis by Demcon TSST in April 2025. Well within schedule for completion of the full research facility, including all diagnostics, in October 2028.

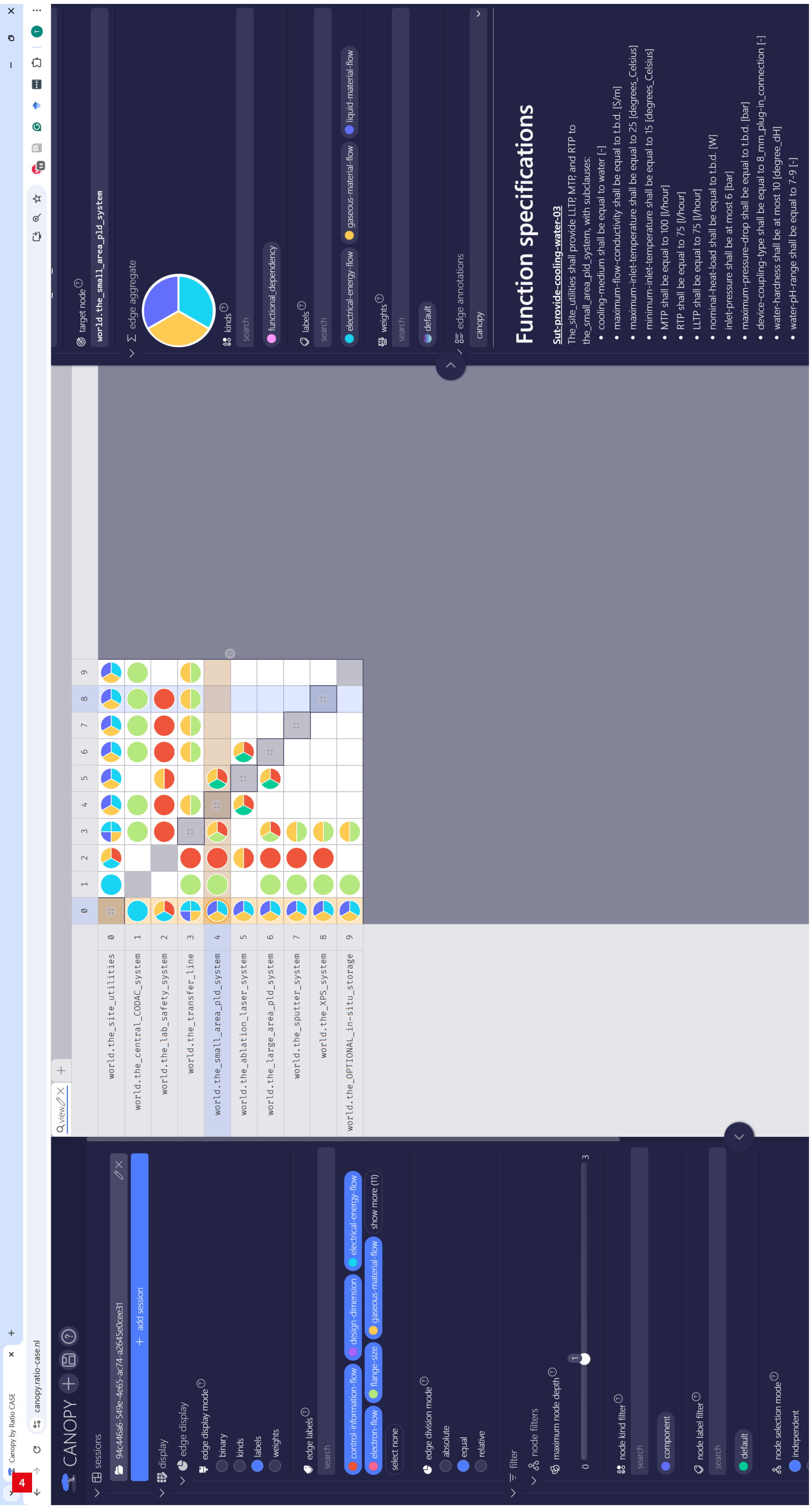
System architecture modelling

A crucial step in the conceptual design phase was creating a structured overview of all hardware and software interfaces between the various components, along with listing the requirements for those interfaces in the form of an architectural model. This model served as the foundation for assessing the impact of design decisions, determining and coordinating the necessary communication between suppliers, and ensuring smooth integration of the respective components later in the design and realisation process.

For example, if two components provided by different suppliers share an interface, the requirements for that interface should be agreed upon with the respective suppliers. Such requirements could be as simple as deciding whether to use metric or imperial tubing.

To create the system architecture model, we used the Elephant Specification Language (ESL), an open-source, computer-readable, natural-language-based language for writing highly structured system specifications. Architecture models are automatically derived from these specifications by the ESL compiler [1,2,3].

A major benefit of using a natural-language-based specification language is that it requires no prior technical knowledge to read, making it easy to review and discuss with researchers. This contrasts with commonly used system architecture modelling languages such as SysML, which are



Function specifications

Sut-provide-cooling-water-03

The_site_utilities shall provide LLTP, MTP, and RTP to the_small_area_pld_system, with subclauses:

- cooling-medium shall be equal to water [-]
- maximum-flow-conductivity shall be equal to t.b.d. [S/m]
- maximum-inlet-temperature shall be equal to 25 [degrees_Celsius]
- minimum-inlet-temperature shall be equal to 15 [degrees_Celsius]
- MTP shall be equal to 100 [l/hour]
- RTP shall be equal to 75 [l/hour]
- LLTP shall be equal to 75 [l/hour]
- nominal-heat-load shall be equal to t.b.d. [W]
- inlet-pressure shall be at most 6 [bar]
- maximum-pressure-drop shall be equal to t.b.d. [bar]
- device-coupling-type shall be equal to 8_mm_plug-in_connection [-]
- water-hardness shall be at most 10 [degree_dH]
- water-pH-range shall be equal to 7-9 [-]

Interactive dependency structure matrix (DSM) visualisation of the PLD research cluster system architecture at decomposition level 1.

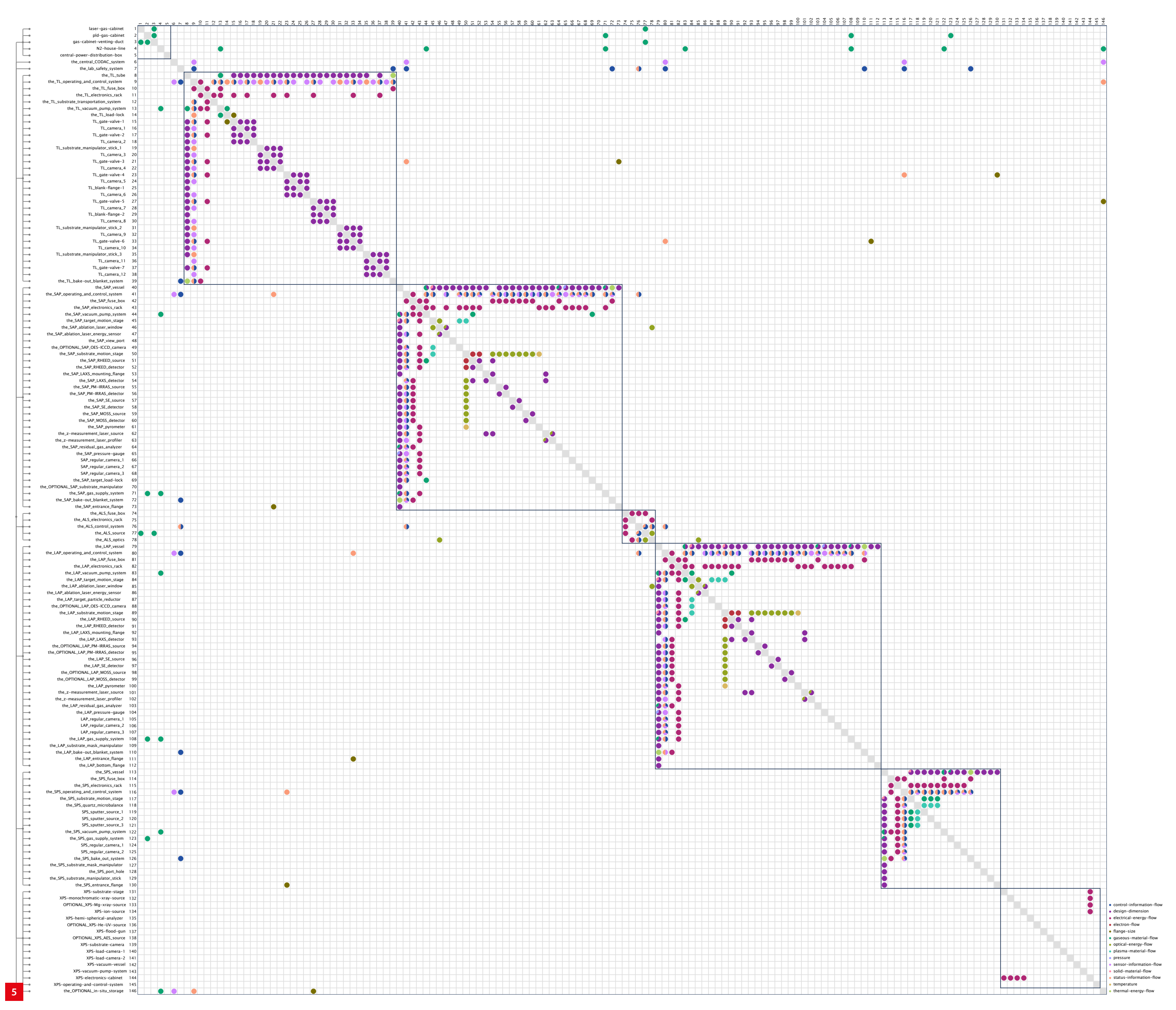
often difficult for non-experts to understand. Additionally, ESL specifications can be easily managed using version-control software, such as Git or SVN.

Listing 1 shows an example goal-requirement specification that states that the ‘site utilities’ shall provide ‘cooling-water-flow-SAP’ to the ‘small area PLD system’ (SAP: small-area PLD), as well as the sub-requirements (subclauses) that shall be met regarding the required pressures, temperatures and device-coupling type. In lines 8 through 10, one can see that ‘cooling-water-flow-SAP’ is actually composed of three cooling water flows: ‘cooling-water-flow-SAP.MTP’ for the main turbo pump, ‘cooling-water-flow-SAP.RTP’ for the RHEED turbo pump, and ‘cooling-water-flow-SAP.LLTP’ for the target load-lock turbo pomp.

From the goal-requirement stated in Listing 1, the compiler directly derives an interface between the site utilities and the small-area PLD system. The whole network of derived interfaces and associated specifications can be interactively visualised in the form of a dependency structure matrix (DSM), as shown in Figure 4. A DSM is a square $N \cdot N$ matrix showing the interfaces among N components in the form of pie charts. For example, the pie chart at row four, column 0 represents the interface between the site utilities and the small-area PLD system. The colours of the wedges in the pie chart indicate the type of interfaces, in this case, being electrical-energy-flow (green), gaseous-material-flow (yellow) and liquid-material-flow (blue) interfaces.

The goal-derived requirements from which these interfaces originate are automatically inserted into the model. By clicking on a pie chart, users can retrieve the associated specifications, as shown in the bottom right of Figure 4. The menu on the left allows users to quickly filter the types of interfaces displayed in the matrix and adjust the granularity level. For instance, Figure 4 shows 10 components and 83 interfaces at decomposition level 1.

By increasing the maximum node depth or expanding a component within the DSM by clicking on the grey diagonal, users can adjust the granularity of the visualisation, thereby increasing or decreasing the level of detail. In total, the PLD research cluster has been decomposed into 146 components,



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Dependency structure matrix of the PLD research cluster showing 146 components and 726 interfaces.

organised across three decomposition levels, with 726 interfaces identified as displayed within the DSM shown in Figure 5. At this level, most components are (semi-customised) off-the-shelf components. Hence, there was no need to further decompose these components and add additional levels of detail.

The compiler automatically derives the interfaces between all components across every level of the decomposition tree based on the specification. As a result, branches of the decomposition tree can be expanded or collapsed arbitrarily while maintaining a consistent overview of the interfaces. This is a major advantage over traditional tools, where maintaining such consistency is typically a manual, time-consuming and error-prone task.

By setting up the model and iteratively expanding and refining it throughout the conceptual design phase, the core team gained a clearer understanding of which parts of the system were well specified and ready for tendering, and which parts remained uncertain and could potentially lead to integration issues.

For example, in-situ LA-EDS in a PLD environment is a novel application still under development. To mitigate uncertainty surrounding LA-EDS, flanges larger than strictly necessary were integrated into the PLD chambers to provide additional space for fine-tuning the angle of incidence of the X-ray detector. Finally, the ESL specifications were used as a single source of truth for generating various PDF and Excel documents detailing the system decomposition and requirements for different subsystems of the PLD research cluster. These documents were utilised in multiple tendering procedures and subcontracts. Because these documents were all generated from the same source, we did not have to check for any inconsistencies between them, which is a major benefit compared to manually creating these documents. For example, it was not needed to check the consistency in components IDs and requirement values across the various documents.

Overall, the extended conceptual design phase and the well-defined system architecture model – with its associated requirements – laid the foundation for a relatively smooth detailed design and realisation phase in collaboration with Demcon TSST.

Of course, not all cost increases and delays could be avoided. For instance, during the conceptual design phase, additional functionality was added to the research cluster that had not been accounted for in the original proposal. The Covid pandemic led to significant inflation and increased lead times. Gas lines leaked, electronics malfunctioned, and software bugs were encountered. However, thanks to the swift start, there was sufficient slack in the schedule to absorb these setbacks.

To the next level

Compared to a nuclear fusion power plant or the Einstein Telescope, the PLD research cluster is a small system. However, applying the same methods and tools to the visual reference spectroscopy system of ITER [4], as well as to the design structure of a plasma control system [5], appears promising.

Moreover, the larger and more complex a system becomes – and the more people are involved – the more critical highly structured interface management becomes. Accordingly, DIFFER, TU/e, and Ratio CASE continue to develop and apply the presented systems engineering methods and tools to enhance scalability, enable distributed implementation for concurrent design and extend functionality.

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Invitation

For more information about the PLD research cluster, its capabilities, associated research programs, the application procedure for access to the user facility or the systems engineering approach, please send an email.

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