Final Report Summary - ISTRESS (Pre-standardisation of incremental FIB micro-milling for intrinsic stress evaluation at the sub-micron scale)

Executive Summary:
The ISTRESS project was firmly based on the triangle of manufacturing, modelling, and experimentation since its original submission. Research and innovation activities where focused on the development of an open innovation environment for the exploitation of high-resolution focused ion beam (FIB) residual stress analysis on a range of industrially relevant materials and devices. Five main actions have been fully accomplished:

1. Development of automated and reproducible FIB residual stress measurement procedures, together with digital image correlation (DIC) software and stress calculation algorithms (WP2);
2. Validation of the FIB-DIC method by comparisons between FIB-DIC experiments and other available high-resolution residual stress measurement techniques, including nano-diffraction, micro-Raman, EBSD,
TEM and Atom Probe Tomography (APT) experiments (WP5);  
3. Quantification of the effects of FIB damage, materials’ anisotropy and microstructure on the strain relief after FIB micro-milling, by means of advanced multi-scale modelling tools (from atomistic, to meso-scale, up to macro-scale), and publication of the results (WP4);  
4. Pre-standardisation of the method, by a series of inter-laboratory exercises aimed at establishing best practice procedures for the realisation of reproducible and reliable experiment, and publication of a Good Practice Guideline by NPL (WP3);  
5. Transfer to industry partners by the design and production of innovative demonstrators with improved in-service performance given by spatially-resolved control of residual stress distribution. Produced demonstrators include novel multi-layer coatings for diesel injection systems (Bosch), as well as micro-devices (pressure sensors and RF micro-switches, Thales).  
At the end of the project, quantifiable results demonstrate the full achievement of the aforementioned goals:  
1. First time for fully automated micro-scale residual stress assessment by FIB on most of materials kind (including amorphous and textured materials); automated experimental procedures have been developed and released on the three main FIB systems on the market (TESCAN, ZEISS and FEI); in order to guarantee higher impact on European industry, maximum priority was given to commercialisation of a TESCAN FIB-DIC software;  
2. An open source code for DIC analysis was developed, optimised and released by the partner Fraunhofer IWM, in cooperation with Oxford university, NPL and Tescan;  
3. As a direct consequence, the method was effectively used for industrial routine control of residual stress, as proven by the achieved performance in terms of time for a single measurement (less than 1 hour) and cost of a single measurement (less than 200 Euros);  
4. First time for quantitative and systematic assessment of FIB induced artefacts as a function of ion-dose and material type, by the publication of multi-sale modelling of ion-matter interaction mechanisms and the effect of elastic anisotropy and microstructure and strain relaxation mechanisms;  
5. First time of direct residual stress assessment on complex 3D micro-electronics devices at a reasonable cost and development of a demonstrator with improved performance (Thales);  
6. First time of residual stress depth profiling in multi-layer hard coatings, and development of a new demonstrator with improved performance (Bosch);  
Dissemination of the results has been proved by the publication of 45 papers in international peer reviewed journals, more than 70 oral presentations and/or posters at international conferences, a dedicated session on this project at the XXX international conference of Surface Modification Technologies (SMT30), the publication of two articles in the most important financial journal of Italy (Il Sole 24 ore), an Industry Road Show (three events in Europe) to demonstrate the new analytical capabilities of FIB-DIC methods. Finally, all developed know-how (including the GPG) will be published in an online Open Innovation Environment, where registered users will be allowed to share their data, to find online tutorials on the technique and to have open discussions with experts in the fields and with enterprises (including SMEs) and other stakeholders.  
The impact of the achieved results on the European industry and society will be remarkable, given the significant increase of product performance that can be achieved by the novel design rules developed in the project, which will be clearly associated to an improvement of the European competitiveness of European products (automotive and microelectronics industry), together with a quantifiable reduction of associated CO2 emissions that will involve clear and measurable societal and socio-economic benefits.
Project Context and Objectives:
The main concept of the ISTRESS project was to develop and promote pre-standardization of an innovative, highly reproducible and automated family of protocols for the measurement and analysis of residual stress at the sub-micron-scale, which affect the properties and lifetime of a wide range of micro/nanostructured and amorphous materials, thin films, MEMS devices and engineering coatings. Research and innovation activities were particularly focused on the development of new characterization approaches and experimental techniques revealing a correlation between local microstructure and residual stress state and the mechanical properties of structured materials at the macro- and micro-scales. In fact, the reliable measurement of residual stress is a critical issue for the robust design and reliability assessment of a wide range of micro- and nano-systems, as they directly affect the adhesion and fracture toughness of thin films, the load bearing capacity, elastic strain to failure and ductility of Bulk Metallic Glasses (BMGs), the reliability of micro-welds and other metal interconnects, crack propagation and charge carrier mobility in semiconductor devices, thermal stresses in through-silicon vias (TSV), the resonant frequency and lifetime of micro/nano-electro-mechanical systems (MEMS/NEMS), the mechanisms of cell adhesion over engineered scaffolds. Despite these urgent industry requirements, the assessment of the residual stress in sub-micron volumes is still an extremely challenging task, especially in the case of nano-crystalline, strongly textured, complex multiphase or amorphous materials and thin films.
To address such extremely demanding industry requirements, the aim of this project was to develop an accessible, reliable and accurate micro-scale residual stress methodology, that can be applied in research studies to support the development of new materials, processes and systems, also allowing routine use in industry, particularly within production and quality control. The methodology is based on incremental focused ion beam (FIB) micro-milling, combined with high-resolution in situ Scanning Electron Microscope (SEM) imaging, a full field analysis of the consequent relaxation strain (or strain relief) by digital image correlation (DIC) and analytical/numerical models for residual stress calculation (fig. 1).

The main focus of the scientific research & development activities was on:
a) the development of automated procedures for residual stress analysis by FIB using different milling geometries;
b) the development of innovative open source DIC codes for strain 2D mapping;
c) the development of innovative calculation procedures for residual stress depth profiling by incremental FIB micro-milling;
d) Development of specific procedures for stress measurements in thin films, amorphous alloys,
e) the development of modelling tools to quantify the extent of FIB damage on a range of materials and its effect on measured strain relief, with the final aim to determine the actual spatial resolution of the technique;
f) the development of modelling tools to quantify the effects of elastic anisotropy and microstructure on measured strain relief;
g) The production of reference materials for evaluation of method’s reproducibility and robustness;
h) The validation of the technique by the use of a wide range of standard and high resolution methods (including curvature, x-ray diffraction, nano-diffraction, micro-Raman, EBSD)
At the same time, the main focus of the industry-oriented innovation activities was on:
a) Development, exploitation at production level of innovative multi-layered nano-coatings with controlled
a) Development, exploitation at production level of innovative multi-layered nano-coatings with controlled residual stress profiles and improved performance. Production of demonstrators;
b) Development, exploitation at production level of novel micro-electro-mechanical-systems (MEMS) with controlled residual stress and improved functional behaviour;
c) Advanced stress analysis on novel polycrystalline alloys, where intra-granular residual stress control would enhance significantly mechanical strength and damage tolerance of aerospace and energy components;
d) Advanced stress analysis on composite materials and biomaterials

The final objective was the establishment of innovative, industry-oriented design tools, implemented via modelling and optimization, for the production of stress-controlled nanostructured and amorphous materials.

Successful achievement of the scientific and technological goals will be accomplished through the exploitation of a synergistic research and development programme between the research and industry partners, which will be based on the following four main concepts:

- Optimization (WP2), validation (WP5) and pre-standardisation (WP3) of a high-resolution method for the spatially resolved residual stress analysis on a sub-micron scale;
- Further insights into the real ion-matter interaction mechanisms and evaluation of FIB induced artefacts, by modelling (WP4) and advanced validation experiments (WP5);
- Direct access for industry, and SMEs in particular, to high resolution residual stress measurement protocols (WP2-WP3-WP6);
- New Design-rules implemented into design-tools (coordinated by industry partners, WP6).

The ISTRESS project was divided into 8 interactive work-packages (WPs), including six RTD WPs, one management WP and one dissemination WP.

WP2 was focused on the development and automation of advanced focused ion beam (FIB), Digital Image Correlation (DIC) and residual stress calculation procedures on a range of materials and coatings that were produced within WP7.

In more details, all of the anticipated goals for WP2 have been fully achieved, as described in the following:

1. FIB automated milling procedures have been effectively implemented on FEI, ZEISS, JEOL and TESCAN FIB/SEM equipment, as described in detail in D2.1 and D8.7;
2. TESCAN, as a full ISTRESS partner, has developed an executable FIB/DIC routine that will become commercialised on TESCAN equipment;
3. A novel ISTRESS DIC freeware has been developed, optimized and published online, as described in the series of deliverables from D2.3 to D2.8. The outcomes of DIC round-robin activities are also reported in D3.7 and D3.8;
4. FEM calculation procedures have been implemented by several partners, as described in D2.9 and D8.7; all possible milling geometries have been analysed by modelling and experiments (hole-drilling, ring-core, single-slot, double-slot, four-slot, and the combination of them).

WP3 was focused on the promotion of standardisation of the technique, by the realisation of Round-Robin activities. In more detail:

1. Stress relaxation measurements on externally loaded samples, in order to evaluate the sensitivity and resolution of the method in case of known stress distribution. All iterations of the associated Round-Robin have been successfully completed (D3.2-D5.5 and D5.6) and the method was fully validated on the
have been successfully completed (D3.2 D3.5 and D3.6) and the method was fully validated on the reference samples;

2. Stress measurements on reference coatings produced within WP7; all iterations of this round-robin have been completed on two different Titanium Nitride (TiN) coatings (fig. 2), and the agreement between measured average stress and the reference values is excellent (D3.4 D3.5)

3. The round-robin activities also allowed to estimate the current strain resolution of the method, which is currently of the order of 5·10⁻⁵;

WP4 activities are focused on MD modelling of the FIB induced damage on a range of materials, and conversion of the calculated damage into an eigenstrain distribution to be used for the evaluation of the effects of ion-damage on the stress relaxation FIB-DIC experiments.

The results achieved in the project allowed to bridge the gap between atomistic scale information on FIB damage and the measured relaxation strains after FIB milling:

1. Final results on atomistic simulations on the FIB-machining process have been distributed to partners involved in the validation activities (D4.3);

2. Using the atomistic simulation as input, the main eigenstrain profile was abstracted and used to evaluate its actual effects on the experiments; this allowed to identify a minimum size of the milled trench, for which the FIB damage can be considered as negligible for a wide range of materials (D4.6);

3. The density, strain and stiffness of the damaged region were successfully measured. Detailed parameter studies and simulations of mechanical tests has been performed;

4. The effects of elastic anisotropy and microstructure of strain relief was analysed in detail, reported to partners and published on international journal (D4.8);

5. Finally, all information was collected into a single and comprehensive analysis on the overall evaluation of the interaction between FIB milling and geometry, anisotropy and the effects of ion damage; results are reported in D4.9 and published online.

WP5 was focused on technique validation and scale bridging and is deeply connected with WP3 and WP4.

The activities have been mostly focused on (a) validation of the FIB-DIC experiments by using independent residual stress measurement techniques and (b) validation of the WP4 modelling activities by using advanced microstructural tools (TEM and Atom Probe Tomography, APT, techniques).

The results achieved (fig. 3) in the project demonstrated a significant advancement in the know-how in the field of advanced residual stress analysis in complex materials and on how to effectively compare results from different techniques:

1. Deliverables D5.1 to D5.6 and D5.13 to D5.15 gave a very comprehensive and advanced overview on how the new FIB-DIC method can be compared with all of the currently available residual stress measurement techniques (curvature, XRD; nano-XRD, micro-Raman, EBSD)

2. TEM and ATP experiments showed that no secondary phases or significant amount of defects are formed after FIB irradiation on the materials and the characteristic FIB ion energy of interest for the project (mostly metals and ceramics: D5.7 to D5.12);

WP6 was focused on the exploitation and transfer to industry partners of the developed FIB/DIC protocols, as well as on the development of novel design rules for production of innovative materials and micro-devices with tailored residual stress distribution and improved performance.

1. Novel modelling tools for designing multi-layer coatings with improved mechanical strength, adhesion and in-service performance were published (D6.1);

2. A series of innovative demonstrators with improved performance were produced at both levels of academic research and industry production (BOSCH D6.2 to D6.4); in particular, the final demonstrator
academic research and industry production (BOSCH, D6.2 to D6.4); in particular, the final demonstrator
produced at Bosch (fig. 4) will be put into production lines immediately after the project;
3. Novel RF-MEMS device demonstrators (fig. 5) were produced at THALES (D6.5 and D6.6);
4. Application of the FIB-DIC method to advanced residual stress analysis in polycrystalline alloys for
energy systems were demonstrated (D6.7);
5. Application of the FIB-DIC method to advanced residual stress analysis on a range of composite
materials, biomaterials and biological tissue was demonstrated as well (D6.8);
WP7 was focused on the development of fully characterised reference samples for method validation, as
well as samples from actual industry production lines.
1. Two different Titanium Nitride (TiN) coatings on Silicon substrate were fabricated, to be used as
reference materials for the FIB-DIC method (D7.1 to D7.3);
2. A series of demonstrators for residual stress testing were fabricated by THALES and BOSCH (D7.4)
3. Efforts will be made after the project to have the TiN materials as established reference materials for
residual stress analysis by FIB and other high-resolution techniques.

Project Results:
In this chapter, all the main &T results/foregrounds developed in the project are described, using the
project milestones as main titles of each sub-section. All figures mentioned in this section are given in the
attachment.

Development of FIB-SEM procedures (MS2)
Semi-automated procedures were developed on the three main systems on the market, namely TESCAN,
ZEISS and FEI FIB/SEM Microscopes. A brief description of the procedures on three systems is as
follows:

Semi-automatic FIB-DIC method for FEI (Roma Tre and ETH Zurich)
The purpose of this software is to provide a simplified tool for the automation of a FIB instrument, with the
aim of implementing the stress measurement methods developed within the ISTRESS project for an
experienced FIB user not necessarily familiar with the FIB toolisation process and FIB/DIC milling
procedures. Assumptions are made that the user is familiar with the FIB instrument and any FIB specific
terminology. Also, it is assumed the user is aware of the outline methodology of the stress measurements
procedures and the specific requirements for the sample being studied.
The method is developed on a GUI and provides user a simple and easy way to generate scripts that can
be use on the FIB system. The software manages all the required parameters for a stress measurement
test, within a simple graphical user interface (GUI). The software has three distinct tabs (fig. 6) namely;
“General Settings”, “Deposition Settings” and “Procedure Settings”. Each panel is activated upon
selection and user can edit/enter the necessary parameters.
The General Settings tab contains the common settings used for both SEM and FIB, such as selection of
ion current, voltage, screen resolution, dwell time, frame integration etc. The Deposition Settings panel is
used to deposit some features on the sample surface using the GIS source (e.g. Pt or C deposition) within
the microscope. It is worth mentioning that while running the FIB-DIC procedure, a decision has to be
made, that whether the sample surface requires some additional markers for the DIC analysis or not? In
case, the additional markers are required, the appropriate pattern images can be loaded into the interface.
The software will automatically package them with the final FIB-DIC script. The Procedure Setting tab is
the most distinctive section of the software. All the milling geometries (e.g. ring core, single slot, double,
the most distinctive section of the software. All the milling geometries (e.g. ring core, single slot, double slot etc.) are available in this section and depending upon the requirement, can be selected from the drop down menu. Upon the selection of desired milling geometry, the necessary parameters (e.g. diameter for ring core, length for single or double slot etc.) are loaded and can be edited as per user requirement. It is also possible to add number of tests on a single sample using this tab. The software has the capability of producing an automated procedure, which consists of multiple parametric-independent sub-test by simply adding the tests as rows. This option allow user to run tests on an extended sample area after the selection of desired target material and beam scan direction. Finally, the “Generate script” button will create a package containing all the necessary information for performing the FIB-DIC procedure. The final script file is a common txt file and can be loaded using the AutoFIB application available with the FEI microscopes.

Semi-automatic procedure for FIB-DIC milling with the TESCAN (TESCAN and OXFORD)

The aim of the developed procedure was to build a dedicated software containing all the steps of the FIB-DIC process from setup, acquisition, data processing and representation of the results. TESCAN AutoDIC is a dedicated software tool developed during the iSTRESS project. The software has been continuously improved based on increasing knowledge coming from the project activities. The AutoDIC software has evolved from a proof-of-concept 3-line script (fig. 7), through a multi-parameter testing tool for Round-Robin test activities into a recipe-based user-friendly tool with rich graphical interface and a distinct data structure (fig. 7).

Sample parameters/description is crucial for selection of proper acquisition and processing conditions. Required sample characteristics/parameters for successful acquisition are: Young’s modulus E (GPa), Poisson’s ratio ν, FIB Milling rate: µm3/nA/s and sample geometry: bulk / layer /depth profile.
The Acquisition Site is a physical position where the analysis is performed with respect to the sample shape and orientation. An acquisition recipe is associated with each site. All parameters of the acquisition and processing are stored together with the site. After the final geometries and parameters were proven, the tool was modified from a parameter oriented tool into a recipe-based tool optimized for typical use-cases according to the Good Practice Guideline (GPG). The Acquisition Recipes, together with known sample characteristics was found to be a necessary approach for successful automation of the acquisition process. The Acquisition Recipes contains: FIB milling geometry describing the geometry used and number of steps (e.g. Ring Core milling with 5 µm diameter pillar should contain at least 10 steps of 500 nm in order to reach the depth required for full stress relief within the pillar), Surface patterning option (e.g. if sample surface is defined as “non-structured”, electron beam deposition should be used to deposit a pattern of dots), Reference mark position and drift correction parameters, SEM image acquisition parameters (e.g. field of view, image resolution, pixel dwell time etc.), FIB milling parameters (e.g. ion beam energy, beam current, spot size etc.), DIC processing input parameters (e.g. image list – generated dynamically, grid of points for DIC – depending on the geometry, image filtering list – predefined for the recipe use case etc.) and Geometry definition for selecting the proper analytical solution for stress calculation.

Semi-automatic procedure for FIB-DIC milling with the Zeiss Auriga 60 (NPL)
The aim of this procedure is to produce a systematic method that can be carried out by an experienced FIB user, but not one necessarily familiar with hole milling for DIC in the FIB. Assumptions are made that the user is familiar with the Zeiss Auriga 60 instrument, the basic functions of the Fibics software and any FIB specific terminology. Also it is assumed the user is aware of the outline methodology of the hole milling
FIB specific terminology. Also it is assumed the user is aware of the outline methodology of the hole milling process and the specific requirements for the sample being studied. Before starting the semi-automated procedure, following steps should be followed:

1/ All materials will have their own unique milling rate. If the material you are working with has a known milling rate then this step can be skipped, but good practice dictates the rate should be checked as aperture wear will increase the ion beam current over time (simple hole milling can do that job ...!)

2/ A decision needs to be made as to whether the sample surface requires additional markers for the DIC analysis. Consider the field of view of the area that the analysis will be carried out over (this is typically a few square µm). Is there sufficient texture for the DIC to track? Or maybe the sample is polished? If in doubt capture a high-resolution image of the sample at the magnification you will use and then move the sample by a small amount (few % of field of view) in any direction and capture a second image. Process these with the DIC software and see if it can track the displacement from first to second image. In case of insufficient features, use the GIS needle and deposit some Pt dots.

Once all the checks have been carried out, the step by step procedure is as follows on Zeiss Auriga 60:

i/ The sample should be tilted to 54° and align and optimise the electron and ion beams on a sacrificial region of the sample near to where the hole milling will be carried out. It has been found that instabilities in the electron beam scan cause significant imaging artefacts if the sample is moved between the image capture and the hole milling process and all should be carried out with the sample tilted normal to the ion beam and without further moves.

ii/ Check focus and astigmatism and adjust the parameters (detector: SESI, brightness (~ 48%), Contrast (~29%), Scan Speed: 3, Resolution: 1024, frame integration N: 16(Time should be approximately 7s): Mag as required (~50kx), Tilt correction ON (54°), specimen current monitor ON

iii/ Set up a folder where the images will be captured (remember to enable automatic numbering of the images for ease of use with the DIC).

iv/ In the Fibics software select FIB and then load one of the pre-set pattern geometries (e.g. ring core, double slot, single slot). In the operations box select “Si marker crosses” (this is an old operation file but has the correct parameters for the hole milling).

v/ In the dose box enter the dose you require for an individual depth increment. Note: this may be for a single hole to full depth, or an increment for depth profiling of the stress.

vi/ For running semi-automatic procedure, open the Zeiss macro “multi image test” in the macro editor and change the values for magnification, contrast and brightness in the macro. Save the macro. Save an image to the chosen folder and number it as “file name”_000.tif where “file name” indicates your choice of name for the sample. This sets up a starting point for the macro to sequentially save images from now on. Set the Auriga mode to EXT and execute the macro by pressing F5 on the keyboard. The SEM image should become live and wait for 30s to allow everything to stabilise before beginning to capture ten images in frame integration mode (16 frames). You can check in the destination folder in real time and see the images arriving there.

vii/ Once the initial images are captured, press run in the Fibics software (making sure the milling pattern is selected) to mill the hole. Once more the patterning window will show the progress of the pattern both graphically and in terms of live and integrated grey-scale images.

viii/ Once hole milling is complete, the sample should appear with a milled hole in the centre of the field of view in the images. If the beams are misaligned and the hole is too far from the centre of the field of view realign the beams on the newly milled hole and go back to point vi, remembering to set a new file name if necessary (it is always prudent to do this).

ix/ Once complete shut down the FIB gun remove the sample and close Fibics and Auriga software. For
Once complete shut down the FIB gun remove the sample and close Fibics and Auriga software. For more details on the procedure, the reader is referred to the “Good practice guide (GPG)” from NPL, UK.

Assessment of Milling Geometries and Implementation of inverse stress calculation procedures (MS1)
In contributing to accomplishing MS1 Assessment of milling geometries and implementation of inverse stress calculation procedures, OXFORD partner used detailed Finite Element simulations to compute the curves for surface strain relief observable by DIC analysis as a function of the milling depth. The results were recorded in parametric form, and described using novel closed-form approximating functions for a wide spectrum of practically important cases. Results were reported to the consortium via D2.2

Implementation of the FIB/SEM procedure, digital image correlation (DIC) analysis and profile fitting.
The subsequent step consisted on the analysis of depth profiling calculation procedures. The development of the Integral Equation (IE) stress analysis technique was presented in D2.9 in conjunction with the depth-resolved FIB-DIC approach. This delivers improvement in the quality of residual stress profile determination by introducing a piecewise linear approximation of the stress profile. The approach was also used for comparative critical assessment of the stress depth profile against laboratory XRD technique.

Our further effort directed at the assessment of different milling geometries and practical approaches has resulted in the elaboration of two approaches that we termed the sequential and parallel milling. The former approach is suitable for stress profiling at the step that exceeds about 5× the outer diameter of the milling feature, to avoid the possibility of interaction between individual markers. On the other hand, if mapping is required at the step size that corresponds to the size of the feature itself, then milling should be undertaken in parallel. In this case, even though interaction between individual features is strong, the initial and final states of deformation are captured correctly, which allows residual stress reconstruction to performed correctly. These approaches have been implemented in a number of specific studies, and reported in a review article that was prepared and published as part of this project.

Implementation of DIC procedures (MS3)
A new Open Source iSTRESS software for digital image correlation has been developed, optimized and released for free to the scientific community:

The project consortium, under coordination of Fraunhofer IWM, has finally achieved an innovative, automated and totally debugged software that was released to the scientific community as a freeware, after agreement of all partners.

The package contains a compilation of .m files for 2D strain and stress measurement from image sequences, that can either run on a Matlab platform or in a compiled version. The individual functions are described in a detailed user guideline that is provided with the software. The software has been validated by running a series of inter-laboratory exercises, in comparison with other commercial DIC routines. Through a series of research and innovation tasks, the software has been progressively improved during the project as reported in submitted deliverables:
• Strategies SEM image noise control and reduction (D2.3);
• Calibration process for the characteristic SEM optical errors (D2.4);
• Novel feature patterns for enhanced DIC (intensity, position, size, shape of physical markers) (D2.5);
• Routine with a graphical user interface (D2.6);
• Alternative ways to improve strain resolution (D2.7);
• increased speed of calculation, precision, correlation quality measures, Ellipsoidal grid, Strain Fitting feature, Image quality checking, displacement smoothing and Silent Mode feature (D2.8)
Run-Time DIC processing
The DIC procedure developed within the iSTRESS project contains a Run Time compiled as a ‘silent mode’ version performing data processing of image data in the background during acquisition. The input parameters for the DIC processing are taken from the acquisition recipe. The DIC processing can be started after the first step of analysis is finished and will remain running in parallel during the acquisition, adding the newly acquired data in real time, monitoring image quality and strain of the observed area during FIB milling. An example of Run-Time processing tool is shown in the fig. 8, from left top: current processed image, correlation coefficient distribution, current image histogram, bottom row: current image average strain x, current image average strain y, acquired strain profile After the acquisition is finished the DIC processing is completed and the strain profile is fitted to the analytical stress-strain function given for each milling geometry.

The final FIB-DIC process output contains strain/depth profiles in X, Y, XY direction, fitted with analytical function for a given geometry. The output also contains calculated values of residual stress $\sigma_x$, $\sigma_y$, $\sigma_{xy}$ in MPa (fig. 9).

Pre-standardisation activities. Improving reproducibility and robustness of the FIB-DIC method (WP3)
Pre-standardisation activities were coordinated by NPL and were focused on three parallel tasks, which are fully achieved at the end of project:
1. Establishment of SEM noise and critical SEM imaging parameters for all available FIB/SEM systems;
2. Fundamental validation of the method by measuring relaxation stress on externally loaded samples;
3. Validation of the method and establishment of reference samples by testing on residually stressed materials;
4. Validation of DIC analysis software
The know-how developed within this WP is finally collected in to an NPL Good Practice guideline (GPG).

Round-robin 1. Stress measurement on externally loaded reference samples (MS4)
The exercise was split in two parts:
RR1.0 - assessment of SEM imaging conditions on a range of Scanning Electron microscopes available in the consortium; this part of RR1 consisted of tests on samples with known ZERO applied stress.
RR1.1 - tests on externally loaded samples by using in-situ bending rigs; this part of RR1 consists of tests on samples with known LINEAR-VARYING applied bending stress.

The motivation for RR1.0 came from the need to benchmark the different FIB/SEM systems used by the partners and to understand how imaging conditions and the stability of the individual machines might affect the quality of the DIC data. Two iterations of RR1.0 were carried out to evaluate imaging and performance of partner FIB/SEM systems. Detailed analyses of over 80 image datasets (from 8 partners) were performed by NPL and Fraunhofer IFW. A set of reference specimens, of a Pt pattern on a Si wafer were produced by NPL, for imaging tests by all partners. The fig. 10 shows representative images of the reference samples showing the variation in quality, noise and resolution from different partner systems (4xZeiss, 2xTescan, 2xFEI, 1xJeol)

A series of tests were carried out under different conditions, capturing 10 images at each stage, which
A series of tests were carried out under different conditions, capturing 10 images at each stage, which were uploaded to the iSTRESS website for DIC analysis by NPL and Fraunhofer IWM. The fig. 11 shows DIC results from images taken after a 20min dwell – a measure of the stability of the different FIB instruments.

The results from RR1.0 enabled the main issues associated with SEM imaging for DIC strain measurement to be identified and significant differences between the available microscopes detected. This allowed the consortium to establish:

• the best SEM imaging conditions for DIC strain analysis
• a series of mathematical criteria for assessing the actual performance of the SEM
• a real-time quality check of the SEM images, that has been incorporated into the automatic iSTRESS DIC routines developed by Fraunhofer IWM and fully implemented by TESCAN

For RR1.1 the FIB-DIC method was further validated by testing on samples with known applied stress. Five partners completed measurements on the nc-Ni and CMSX-6 samples, using a special miniature in-situ bend rig designed by TUDA/Erlangen University (fig. 12). Procedures and geometries were agreed with input from WP2 and results from the FIB-DIC analysis was then compared with the analytical Euler Beam theory and FE modelling of the resulting stresses during 4-point bending. The Figure 12 shows the TUDA-designed miniature in-situ bend rig and representative NPL data from tests on the CMSX-6 super alloy, taken at different positions across the loaded beam. The FIB-DIC method was fully validated and the results below show a clear and symmetrical transition from positive to negative relaxation strains made on the compressive and the tensile regions of the bar.

Conclusions from RR1.1 work show:

• A range of milling geometries and bend rigs were used successfully by the different partners
• The measured stresses were in good agreement with the externally known applied loads and model predictions.
• Analysis of the data showed that the strain resolution for the DIC procedure is of the order 10^{-4}.
• Results have been used to optimise the FIB-DIC test protocol and the in-situ test has been incorporated into the Good Practice Guide as an important validation exercise.

More detailed results on this work have been reported in Del 3.2

Round-robin 2. Tests on residually stresses materials (MS5)

This work focused on optimizing the experimental procedures for milling and imaging and, in doing so, compared the performance of the different FIB systems and approaches taken by the partners. Ten partners - NPL, UniRoma3, FAU Erlangen, Fraunhofer IWM, TESCAN, Oxford University, Thales, TUDA, Fraunhofer ENAS and Fraunhofer IKTS – have contributed to this work, ensuring that all 4 FIB manufacturers - TESCAN, Zeiss, FEI and Jeol - were represented.

In 3 iterations, carried out over the first 2 years of the project, over 40 tests were completed and 8000+ images analyzed. Most of the work was carried out on the C01 TiN coating, produced by Daniel Rostislav at Leoben. The round robin exercises have proved invaluable for developing good practice and benchmarking performance – between partners and for individual partners to refine and develop their local test procedures.

By the 3rd iteration, a detailed test protocol had been established, leading to significantly improved results, both in terms of consistency between the different partners and the reduced uncertainty in the strain measurements. Fig. 13 show the excellent consistency of the ring core and double slot geometries milled in the TiN coating by different partners, and the quality of the representative strain relief data obtained.
Due to the large number of experiments and the range of different setups and equipment used, the results in this part of the work have been invaluable in both sharing good practice and identifying issues that have arisen from the work.

Conclusions from the RR2 activity show:

• Throughout the exercise the quality of the results has improved with each iteration, although controllable uncertainties and variability in the measurement still exist.
• The quality of data between partners and FIB instruments varies significantly, particularly regarding the quality of the images captured, which is still probably the most important factor.
• Some partners have been able to semi-automate the FIB milling and imaging process and this is an excellent solution for reducing uncertainties and making the FIB-DIC procedure more commercially attractive.
• Some FIB systems appear to be inherently more stable and perform better and this will have a direct impact on the quality of the DIC data and measurement result.
• Based specifically on this work robust protocols for carrying out the FIB-DIC milling and DIC analysis of the images have been developed and incorporated directly into the Good Practice Guide.

More detailed results on this work have been reported in Del 3.4 and Del 3.5.

Round-robin 3. Validation of DIC analysis software (MS6)
A number of image sets generated in the WP3 round robin exercise on residually stressed materials were identified for the DIC software validation exercise, covering the Ring Core (fig. 14) and Double Slot geometries (fig. 15) from different partners and FIB systems: Sets 1 and 2 were chosen for the initial exercise.

Seven partners were involved in the exercise - NPL, UniRoma3, FAU Erlangen, Fraunhofer IWM, TESCAN, Oxford University, Fraunhofer ENAS and Fraunhofer IKTS. Partners were asked to analyze the image sets using the iSTRESS DIC software to compare and examine the effect of different users and parameter settings. The images were also analyzed by NPL using the LaVision software and Fraunhofer ENAS using the VEDDAC software for comparison. The fig. 16 below show representative figures of the analyses of the different image sets – plotted as relief strain vs image number - highlighting the typical uncertainty in the measurements due to different DIC parameters and analysis settings.

A second iteration was carried out using more specific instructions, and this generally led to reduced uncertainty and scatter. Some of the key issues identified from the RR are summarized below:

• Image quality is still probably the most important factor for reducing uncertainty in the strain measurement. If good quality images are available, the DIC processing is more stable and robust and less sensitive to the DIC parameters used, and any subsequent filtering and cleaning operations.
• Generally, there was good agreement between the IWM, NPL LaVision and Fraunhofer VEDDAC software.
• Averaging of images gives more robust results, with reduced scatter and uncertainty.
• Care is still needed to ensure that the results are not significantly affected by the operator, particularly regarding the area over which the processing is carried out and the DIC parameters.
• Even with a good dataset and images, results indicated that the typical uncertainty in the maximum strain relief due to operator input and variations in processing conditions could be as high as 5 × 10^-4.
• This gives some bound on the likely uncertainty due to the DIC analysis itself.
This gives some bound on the likely uncertainty due to the DIC analysis itself. Additional contributions associated with the practical aspects of the FIB-DIC approach, such as the stability and performance of the particular FIB system, imaging conditions, milling induced damage and artefacts and operator experience should be considered and included in the uncertainty budget.

- For the iSTRESS DIC software, shift correction, averaging and filtering of the images before DIC generally yielded some improvements; cleaning and averaging after DIC was also beneficial.
- For the iSTRESS DIC software cleaning by standard deviation and distance thresholding leads to more stable results compared to cleaning by neighbor movement.

The outputs and findings from the work have been used directly to optimize the FIB-DIC test protocols and incorporated directly into the Good Practice Guide. More detailed results are present in Del 3.8.

Modelling of FIB-induced artefacts and the effect of inhomogeneities and anisotropic elasticity (WP4)

The goal of WP4 was to obtain information on the theoretical limits of the FIB-DIC method. To this end, the FIB-milling process and the created artefacts were studied by atomistic simulation methods. FEM and CP-FEM were used to model these effects based on input from the atomistic simulations and experiments, as well as to assess effects of pre-existing strain in-homogeneities and elastic anisotropy. All anticipated milestones were fully achieved, as reported in a series of publication in high-impact international journals.

Atomistic modelling of FIB damage (MS7)

This milestone is fully accomplished by the realization of these deliverables: cascade simulations (D4.1) atomistic simulations of FIB-machining process (D4.2) and extraction of information for continuum modelling (D4.3 D4.6) as well as comparison with experiments (D4.3).

The initial focus of this milestone was to set up, test and validate the simulation processes involved in the atomistic simulations of ion irradiation, in particular the critical phenomenon of collision cascade. This work summarized in the deliverable D4.1 involved the definition of a proper simulation setup and comparisons of Monte Carlo simulations with atomistic simulations performed within the iSTRESS project or available in the literature.

A crucial part of this milestone was the simulation of the focused ion beam (FIB) machining at the atomic scale. It was successfully achieved by using high performance computing facilities and a specific sample geometry. Unexpectedly, the results shown a close interplay between internal strain and irradiation induce damage. Detailed analyzes of the physics at the origin of the formation of the defects revealed a stronger impact of this dependency in semiconductors than in metals. The need of accurate interatomic models in order to efficiently represent the formation of irradiation induce damage has been also highlighted. In addition, a novel method for determining the key property at the root of atom sputtering has been proposed. This work has been published in two peer-reviewed international journals [1,2].

The most challenging aspect of this milestone was about performing atomistically informed continuum modelling of irradiation induced stress/strain. In other words, how to transfer quantities from atomistic simulations towards higher scale models, like the finite element method (FEM). Atomistic simulations where used to generate irradiation induced damage and to calculate the stress distribution at the atomic scale. This data where homogenized, both in space and time, and used to fit a stress profile usable in FEM. Mesoscale simulations has been thus performed using atomistically informed FEM and published in a high quality journal [3].

In the framework of the iSTRESS consortium, a reliable comparison of the simulations and models with experiments was a crucial requirement. In this milestone, that was achieved in two different ways involving three partners (FAU, UOX and ETHZ) over two work packages (4 and 5). The comparison of atomistic
three partners (FAU, UOX and ETHZ) over two work packages (4 and 5). The comparison of atomistic
simulations with experiments has been carried out using atom probe tomography (APT) experiments on
silicon samples. The experiments consisted on a careful irradiation of the samples and subsequent
analyses using APT. For the first time, atomistic simulations of collision cascades were performed on
samples directly informed by APT data. While validating the modeling approaches chose in the project,
these comparisons raised fundamental questions regarding proper atomistic simulations of ion irradiation.
A second comparison with experiments was achieved by using the FEM as described above. The
deflection of an irradiated cantilever was measured experimentally, and compared with the deflection as
obtain by atomistically informed FEM simulations. An excellent match was reported in an article published
in peer-reviewed international journal [3].


Effects of elastic anisotropy on stress relief (MS8)
This milestone is fully accomplished, in particular by the realization of the deliverable D4.7 and the
submission of the results for publication [1].
Elastic anisotropy can have a significant effect on the reliability and precision of residual stress evaluation,
since the stress evaluation involves the tensor multiplication of the elastic constants by the measured relief
strains. For the Focused Ion Beam – Digital Image Correlation (FIB-DIC) ring-core method, a
Mathematica© package has been developed that allows the evaluation of the complete in-plane residual
stress state from the measured strain relief values using known material orientation and anisotropic elastic
properties for materials with arbitrary elastic symmetry. This makes use of the symbolic manipulation
capabilities of Mathematica© to obtain explicit expressions for the anisotropic stiffness matrices in the
conventional Voigt notation. Furthermore, for any specific material symmetry and properties these
expressions can be exported into a computable form suitable for the use in Matlab© or Fortran (e.g. for
incorporation into FE calculations), containing the three Euler orientation angles as parameters. With the
help of such code, correct evaluation of stress is possible for any materials with known elastic properties
and orientation that can be determined locally e.g. using EBSD. Taking the capabilities of this approach
further, we noted that in many practical situations the underlying material orientation is unknown, so that
only the nominal isotropic continuum elastic constants can be used. This uncertainty leads to an error in
the stress evaluation. We therefore carried out statistical analysis of the uncertainty in stress evaluation
that arises due to the unknown material orientation. We considered materials with cubic crystal and elastic
symmetry that possess different degrees of elastic anisotropy, since for this class of materials it can be
conveniently expressed by a single parameter (Zener anisotropy ratio). With the help of this procedure we
were able to quantify the uncertainty arising from the underlying anisotropy of elastic properties, and to
demonstrate the application of this procedure to a real case of micro-scale residual stresses in a nickel-
base super alloy.


Effects of elastic-plastic inhomogeneities on stress relief (MS9)
This milestone is fully accomplished by the realization of these deliverables: internal project report on
eigenstrain-FE approach (D4.4) effect of FIB-induced eigenstrain on the apparent surface strain (D4.5)
abstracting eigenstrain profiles from atomistic simulations (D4.6) and effects of elastic and plastic
inhomogeneities on stress relief (D4.8) [1-3].
Real materials are never completely free from residual stress and residual deformation that arises from prior processing, sample preparation for examination, and even from the stress evaluation procedure itself. For example, Focused Ion Beam milling that is involved in the FIB-DIC procedure is known to induce ion implantation, lattice defect generation, amorphisation and residual stress modification that we have quantified for the case of crystalline silicon subjected to FIB milling using Ga+ ion beams.

The effect of pre-existing elastic and plastic inhomogeneities of stress on the perceived stress relief was studied experimentally by considering a number of cases where such effects could be introduced in a controlled manner. Shot peening is a procedure for surface treatment that introduced residual stresses by inducing plastic deformation in a confined layer. We studied the nature of the residual stress around notches of different geometry that are associated with different degrees of stress concentration, and found that eigenstrain provided a suitable generic basis for the description of these phenomena.

In a further study we considered the effect of elastic-plastic inhomogeneities at the grain level. When tensile stress applied to a grain agglomerate is sufficient to cause plastic deformation, yielding proceeds differently in different grains, depending on their orientation, shape, neighbors, etc. The amount of plastic deformation and the associated residual stress is determined by both the elastic and plastic anisotropy effects. In order to reveal and quantify the attendant mechanisms, we subjected an aluminum alloy polycrystal to four-point bending beyond the elastic limit, and evaluated residual stresses in individual grains. We were able to reveal and quantify the amount of deviation of the residual stress from the nominal continuum value. These findings serve as a basis for improved predictive design methods for against fatigue and creep failure, for structural integrity in energy applications.

In D4.7 (see MS8 description) an analytical procedure using the Mathematica© package was developed, to evaluate the residual stress using appropriate formulation of the anisotropic stiffness matrix. In addition, statistical analysis for cubic materials with different anisotropy factors was performed, aimed at evaluating the uncertainty that may arise when the material local crystal orientation is unknown, in FIB-DIC measurements.


Validation activities – bridging scales among different residual stress measurement techniques (WP5).

The goal of WP5 was to perform cross-validation of the FIB-DIC method with other local and integral or macroscopic techniques for residual stress characterisation. During the project, the comparability and reproducibility of the results obtained by the FIB-DIC method were fully addressed, critically analysed and discussed.

Validation of the method by synchrotron X-ray nano-beam diffraction analysis of residual stress (MS10)

A position-resolved cross-sectional synchrotron X-ray nano-diffraction approach operating with focused beams below 100 nm allows in principle to characterize nano-crystalline thin films with a grain size down to ~5 nm and to reveal large-scale lateral homogeneity and non-homogeneous depth gradients of structure, strain and properties typical for most of the technologically relevant nanostructured thin-film materials. The advantage of the new scanning method is that the depth gradients of microstructure, residual stresses and phases can be determined directly in real space as a function of the coating depth z. This has an enormous technological and practical importance as it allows to access the full picture of microstructure and strain distributions across the thin film depth with very high resolution given by the beam size.

The ISTRESS reference TiN samples (C01, C02, WD51 and WD52) with specific stress profiles were
The ISTRESS reference TiN samples (C01, C02, WD51 and WD52) with specific stress profiles were characterized at the nanofocus extension of the ID13 beamline at the European Synchrotron Radiation Facility in Grenoble, France. The experimental setup is presented in fig. 17. A slice of the film with the thickness \( L = 50 \mu m \) in the beam direction was scanned at \( E = 13 \) keV in transmission geometry using a beam oriented parallel to the film-substrate interface. The thin sample slice was aligned with the film/substrate interface oriented parallel to the beam (fig. 17) by using the \( \phi \) axis. For each position a charge-coupled device (CCD) area detector with a resolution of \( 2048 \times 2048 \) pixels and a pixel size of \( 50 \times 50 \mu m \) positioned behind the sample at the distance of 9.2 cm collected a Debye-Scherrer diffraction frame at a counting time of 0.5 s per frame. For the evaluation of the film microstructure properties from Debye-Scherrer rings, it was necessary to use an X-ray beam with a relatively large full width at half maximum (FWHM) of 250 nm to obtain sufficient grain statistics along the TiN (111) and (200) rings (cf. continuous rings in fig. 17).

In the case of strain mapping, the aim was to achieve higher spatial resolution. For that purpose, a beam with an FWHM of 100 nm was applied. The scanning experiments were performed by translating the sample in the beam along the \( z \)-axis with steps matching the applied beam sizes of 250 and 100 nm. The characterization of fibre texture is trivial in this case, including also full orientation distribution function calculation, especially in the case of in-plane isotropic thin films with a fibre axis oriented perpendicular to the substrate surface as no Laplace transformation is needed as in the case of diffraction in reflection geometry. The two-dimensional (2-D) diffraction data were treated using the program package Fit2D, especially developed for the evaluation of the nano-diffraction data.

The synchrotron XRD studies carried out in ISTRESS provided the complete quantification of the gradients of texture, phase composition, peak profile and strain across the thickness of selected reference nano-crystalline thin film samples, namely C01, C02, WD51 and WD52, as revealed by synchrotron position-resolved X-ray nano-beam diffraction analysis in transmission geometry (e.g. fig. 18 for the stress profile of C02). The data were delivered to all project partners as complementary results for the development and validation of the FIB-DIC method and for improvement of the resolution of the method to determine stress profiles by applying new approaches such as data acquisition of a series of ring cores with various diameters.

By realizing deliverable 5.1 reporting the experimental results on the selected reference samples, the milestone M10 “Validation of the method by synchrotron X-ray nano-beam diffraction analysis of residual stress” of the ISTRESS project has been fully achieved.

Validation of the method through the combination of \( \mu \)-Raman, EBSD and macro-scale measurements (MS11)

The aim was to use \( \mu \)-Raman, EBSD, and macro-scale measurements (mainly the curvature method) to create reference strain-stress values for the ISTRESS samples, to get a deeper insight into strain release in the FIB-DIC procedure, and to study the influence of the FIB irradiation.

Micro-Raman analyses of stress states present e.g. in diamond coatings were performed and compared to FIB-DIC measurements and finite element analysis. Furthermore, a detailed analysis of Raman peak shifts in the region of different FIB-DIC geometries was conducted. An overall good agreement between FEA, FIB-DIC and Raman analysis was observed (fig. 19). In summary, it can be said that Raman spectroscopy enables direct imaging - at least in a qualitative manner - of stress states in silicon and diamond coatings with high spatial resolution and is therefore a helpful tool for deeper understanding of stress relaxation processes during FIB-DIC and for improvement of the different milling geometries used within the ISTRESS project. Nevertheless, quantification of the residual stress values from a single peak shift is of
One purpose of the Electron Back Scattered Diffraction (EBSD) studies in ISTRESS is the analysis of the degree of material amorphisation as a consequence of FIB irradiation. By means of EBSD this is possible by capturing the underlying crystal quality and orientation by probing a shallow material layer achieving sub-micron spatial resolution. The target material is illuminated by an electron beam that undergoes diffraction, producing a so-called Kikuchi pattern. The degree of material crystallinity affects the quality of such Kikuchi patterns, so that the pattern quality is progressively reduced, and in the extreme case of fully amorphous material the pattern is entirely destroyed. The study is devoted to determining the extent of ion beam damage and the effect on the target microstructure at the nano-scale. This is achieved by quantitative assessment of the quality of Kikuchi patterns that can be correlated with the degree of material amorphisation. Target materials (here Si) were exposed to several distinct Ga-ion doses, and the loss of Kikuchi pattern quality was evaluated (fig. 20).

The study has shown the capability of EBSD technique concerning the analysis of material surface amorphisation. EBSD was shown to be suitable for qualitative assessment of the loss of material crystallinity / material amorphisation following exposure to Ga-ion beam. The analysis of a single spot exposure showed that the effect of beam “tail” is increasingly significant at high ion doses.

Further studies were performed on the surfaces surrounding FIB-DIC markers and were used for displacement and strain analysis in the regions where the effect of the ion beam was prominent. Indeed, a high degree of amorphisation was observed in the vicinity of milled edges (fig. 20). In addition to material amorphisation, changes in the surface morphology may lead to significant error in DIC displacement analysis. The evidence collected in this study confirms the appropriateness of excluding from DIC analysis the regions strongly affected by ion beam damage and material amorphisation.

For the macroscopic measurement of the stresses in the ISTRESS thin film samples further used for comparison to the results of the local methods, the curvature method was chosen. Stress in a thin film on a flexible substrate induces a curvature of the substrate. If the substrate is much thicker than the film the induced deformation will be small and purely elastic. By measuring this curvature and knowing the elastic constants as well as using Stoney’s equation the (average) intrinsic stresses of the thin film becomes accessible.

Comparing the results of the different techniques on the same samples it was observed that all the measurements agree on the magnitude of the stress inside the films. However, FIB measurements seems to amplify a bit the stress in respect to the corresponding curvature measurement, making tensile stress more tensile and compressive stress more compressive. The XRD stress measurements conversely are more accurate, i.e. no bias in the evaluation.

By realizing deliverable 5.2 5.3 5.4 and 5.14 reporting the experimental results on the selected reference samples, the milestone M11 “Validation of the method through the combination of μ-Raman, EBSD and macro-scale measurements” of the ISTRESS project has been fully achieved.

In-situ experiments for providing a reference stress state on selected samples and geometries (MS12)

The objective was the design of an in-situ SEM four-point bending device and its use on ISTRESS samples (nano-crystalline nickel and a single-crystal Nickel super-alloy) in the partners focused ion beam (SEM-FIB) systems to test the reliability of the FIB-DIC procedure. The finished design was delivered to the ISTRESS partners who then used the device in their SEM-FIB systems (fig. 21).

The bending apparatus was used to introduce a uniaxial stress state on different reference samples. The 4-point bending has the distinct advantage that controlled stresses (compressive and tensile) can be...
Point bending has the distinct advantage that controlled stresses (compressive and tensile) can be introduced in the material. The results of FIB-DIC analysis are then compared with the analytical Euler beam theory for the stresses during 4-point bending. The linear variation of applied load during bending enables a controlled range of applied stresses to be generated in a single sample. Using this method, a wide range of FIB milling geometries can be tested with the aim of generating purely uniaxial relaxation, enabling simple and robust estimates of the residual stress using Hooke’s law, and the evaluation of the sensitivity of different geometries and methods. The present report summarizes the residual stress measurements using the two slot (H-bar) FIB milling geometry.

The results of the experiments compared to the calculated stress values for two samples are shown in fig. 22 (here as an example isotropic nc-nickel and anisotropic CMSX-6 super alloy). For both samples, the bending stress gradient could be measured accurately, despite an offset in the gradient for the CMSX-6 sample. This offset can be explained by plastic deformation during sample preparation or the bending. In conclusion, the conducted double slot- or so called H-bar-geometry provides a linear and symmetric displacement gradient, yielding reliable residual stress measurements with FIB milling and DIC analysis. This semi-automated procedure is a standard FIB milling procedure and hence several measurements can be performed in a very short time. The values obtained via the FIB-DIC procedure are in accordance with the results obtained by the Euler beam theory. Finite element simulations are required to verify these values, because this sample is expected to exhibit anisotropic behaviour and the simple use of Hooke’s law may be not sufficient for a correct stress estimation. By realizing deliverable 5.5 and 5.6 reporting the experimental results on the selected reference samples, the milestone M12 “In-situ experiments for providing a reference stress state on selected samples and geometries” of the ISTRESS project has been fully achieved.

In-situ TEM on ion irradiated materials and/or FIB irradiated materials (MS13)

Here, the main task was to study the influence of the FIB-ions on the irradiated materials. Sub-tasks were e.g. the determination of the size of the FIB-ion affected zones, the determination of FIB-ion range vs. ion energy, the determination of the formation of secondary phases, and the determination of the predominant defect kind. To reach these goals -amongst others- SRIM (Stopping and Range of Ions in Matter) simulations as well as TEM (Transmission election microscopy) and the atom probe experiments have been conducted.

The experiments show that no secondary phases are formed after FIB-sample interaction, at least for the materials in ISTRESS. This is a relevant conclusion, because we may infer that no additional relaxation strains due to phase transition could be expected after FIB milling for residual stress assessment. For example, fig. 23 shows the analyzed TEM lamella and an atom probe tip of the BMG. From this TEM analysis it can be shown that the BMG is not fully amorphous, but no secondary phases or defects could be identified.

Further experiments show that gallium contamination is the main kind of lattice defect that can be found after FIB milling. No other defect formation mechanisms have been observed, at least for the materials that have been investigated in ISTRESS. Fig. 24 shows a TiN (C01-24) atom probe tip that has been mounted on a TEM grid. The mass spectrum of the sample with two Ga peaks is shown on the right.

In fig. 25 (left) the Ga distribution derived from a Ga-irradiated atom probe sample is plotted. It is clear that the Ga concentration is highest on top and on one side. To define the Ga content, it is necessary to define a specific region that is used to determine the relative Ga content. The Ga content from the surface to the middle region and the according cylinder are shown in fig. 25 (middle + right). Directly at the surface relatively high Ga contents were observed, while the Ga content drops to less than 1% at a distance of 10...
Relatively high Ga contents were observed, while the Ga content drops to less than 1% at a distance of 10 nm from the surface.

In order to relate the ion range to the ion energy, SRIM (Stopping and Range of Ions in Matter) calculations have been performed. SRIM is a Monte Carlo computer program that calculates interactions of energetic ions with amorphous targets. The implantation depth increases with higher energy. While the implantation depths are similar for Al and Si, around 25 nm at 30 kV, the depth is much smaller for Cu, around 10 nm at 30 kV. For low energies the implantation depth of Xe is slightly larger than for Ga, while it is significantly smaller at higher energy levels. The incidence angle is relevant for the application of the ISTRESS method as the milling procedure reaches the analysed surfaces at an angle close to 90° when pushing the ring structure further down. Therefore, the impact of the incident angle of the ions is analysed. The result is summarized for Ga in fig. 26. This is a significant basis to be used in modelling activities for improving the understanding of ion-to-matter interaction mechanisms during FIB experiments.

By realizing deliverable 5.7 5.8 5.9 and 5.11 reporting the experimental results on the selected reference samples, the milestone M13 “In-situ TEM on ion irradiated materials and/or FIB irradiated materials” of the ISTRESS project has been fully achieved.

Analysis of elastic properties of all investigated samples materials (MS14)

For the FIB-DIC method, the in-plane relaxation of the sample surface due to FIB induced strain-stress release is used to determine the horizontal stress state. Most of the samples studied within the ISTRESS project are thin film systems. Since the thin films are mostly anisotropic (transversal isotropy), the in-plane elastic properties are required. Nanoindentation is a method to achieve average values over all spatial directions. In case of FIB-DIC, surface acoustic wave (SAW) method a possible alternative approach. This method gives the in-plane elastic modulus and, if the sound transmission is good, Poisson’s ratio ν and mass density of the sample can also be evaluated. SAW measurements have been carried out for the samples accessible. The results were then compared to nanoindentation results and best values for the stress determination have been chosen.

To all the samples the mentioned measurement techniques have been applied. The recommended modulus values for the determination of stresses from the strains in the FIB-DIC procedure are marked in bold red. The criteria for this selection are: If SAW was applicable, this is the best method for the FIB-DIC procedure and the values should be used. The second choice method, if applicable, is the elastic spherical nanoindentation followed by elastic-plastic nanoindentation.

By realizing deliverable 5.15 reporting the experimental results on the selected reference samples, the milestone M15 “Analysis of elastic properties of all investigated samples materials” of the ISTRESS project has been fully achieved.

Innovation activities: effective transfer to industry of the novel FIB-DIC method (WP6).

As befits a project aimed at establishing a novel technique with wide ranging capabilities, through Round Robin exercises, formulating Good Practice and running a series of feasibility studies, iSTRESS activities included the identification and demonstration of the method in application to a wide variety of problems of great practical significance.

The crucial advantage of FIB-DIC over prior state-of-the-art can be formulated concisely as:

FIB-DIC allows evaluating the local absolute residual stress state, including the full in-plane tensor and the variation with depth below the sample surface, with the accuracy down to single figures of MPa and at the spatial resolution from 10µm down to ~50nm and better.

Based on this mission statement, we identified a range of significant challenges and unresolved problems.
Based on this mission statement, we identified a range of significant challenges and unresolved problems in materials science and engineering where the application of FIB-DIC can bring crucial insights that can help make breakthrough progress towards new understanding and disruptive technologies. We classified the range of applications into a series of broad categories: (i) coatings and multilayers that are obtained by careful deposition that allows intricate control over the nano- and micro-structure, and residual stress (including those for mechanical and micro-electronics applications); (ii) stress concentrators (e.g. notches, fillets) and cracks, including those grown under fatigue conditions and subjected to complex loading history; (iii) advanced polycrystalline alloys for energy applications, including materials and components fabricated by additive manufacturing (AM) and powder metallurgy routes; and (iv) bio-composites, including complex hierarchical natural mineralised tissues, such as human dentine and enamel, as well as interfaces and gradient transition layers.

New product design rules for architecture design of multi-layered coatings by sub-micron resolution (MS15)
The objective of Milestone (MS) 15 is A) The delivery of an improved algorithm for multilayer coating design, based on spatially resolved residual stress analysis and B) Production and characterization of specific innovative multilayer coating systems.

Two different coating design approaches were taken to achieve the objectives of MS 15. The first one is based on finite element modelling (FEM) routine running in the commercial software ANSYS (fig. 27). With the help of this tool optimization of multi-layer coating architecture is possible by the minimization of interfacial residual stress. The tool can simulate the stress distribution arising from the PVD process and then find the optimal layer distribution for having the minimum interfacial stress and hence, increasing the adhesion of the coating.

The second tool was implemented within the existing analytical software package “FilmDoctor”. This is a purposely developed intrinsic stress optimization module (fig. 27). The optimization algorithm works in a way of reducing a given stress component (or even the von Mises equivalent stress), while optimally distributing, by iterations, the intrinsic stress profiles within the coating thickness. To validate the multilayer coating design algorithms within ISTRESS project, a number of case studies were carried out.

In the first case, titanium-titanium nitride (Ti-TiN) multilayer system was studied within the ANSYS routine (fig. 28), to find the optimal thickness of individual layers in a multilayer that can decrease interfacial axial and in-plane shear stress. The coating configurations were experimentally produced at Roma Tre and were quantitatively accessed for their scratch adhesion, in-plane residual stresses, as well as hardness and elastic modulus. The results show that the multilayer in comparison with bi-layer shows significant improvement (22%) in adhesion under decreased interfacial stress conditions without an effect on overall coating stiffness and hardness. The multilayer coating in comparison with different configurations shows an increase scratch adhesion of 18% and 27% for the optimal position and thickness of interlayers respectively. All these results were published in the Journal of Materials & Design (section 4.2) and clearly demonstrate that the approach taken could be useful to develop stress-optimized coatings for wear resistance applications.

In the second case, automated optimization algorithm within FilmDoctor was used to determine the desired residual stress through-thickness profile for a range of contact loading situations (fig. 29). On the basis of modelling activities, three different chromium-chromium nitride (Cr-CrN) multi-layers were produced at Roma Tre, with the aim of obtaining different stress gradients, while keeping same microstructure, the same average stress value and same average hardness in the coating. Results show a significant correlation between the observed residual stress profiles and scratch adhesion, where different
Significant correlation between the observed residual stress profiles and scratch adhesion, where different optimal stress profiles are identified for different loading conditions. This is actually a major step with respect to the previous literature, where scratch adhesion in hard coatings was only correlated to the average stress in the film, but not to the stress gradient within the film thickness. Results shows, that a lower interfacial compressive stress and a reduced through thickness stress gradient gives improved scratch adhesion, when using 10 µm and 200 µm sphero-conical indenters. The outcomes of this activity was also published in the special issue of the Journal of Materials & Design (section 4.2). The results of these studies clearly demonstrated that the tools developed within ISTRESS project are useful and new coating architecture can be designed as well as it is possible to produce coatings with tailored residual stress profile.

The applicability of these tools were further extended with the help of the industrial partner Robert BOSCH GmbH to address a key design problem related to diesel injection systems. In the automotive industry, micro-particle damage is a key failure mechanism in diesel injection systems. The wear and friction arising from the entrainment of micro-particles into fuel under extreme loading conditions or due to imperfect filtering cause valve leakage, lower fuel injection pressure and engine performance and increase particulate emission from the engine. Using the analytical algorithm developed within the ISTRESS project, this key failure mechanism was analytically modelled. Based on the simulation results and backed with extensive characterization of through thickness residual stress profiling (FIB-DIC method), number of demonstrators were designed and produced at BOSCH using as benchmark their proprietary coatings. These demonstrators were then subsequently characterized for their mechanical and tribological properties and results show excellent enhancement in the adhesion of new demonstrators. The last two demonstrators, designed after a careful study of the first three prototypes, show performance increases between 50% and 150% of the best samples of the first iteration (fig. 30). This result highlight the potential of the coating optimization protocol devised in the ISTRESS project for all companies developing wear protection films optimized for a specific mechanical component.

These results show that the design of novel multi-layer coatings with dedicated cross-sectional microstructure, stress and functional design is possible with the help of the tools developed within the ISTRESS project. This is a major step with respect to the current literature, clearly indicating that by fine tuning the coating architecture while guiding their microstructure and properties, considerable enhancement of different properties can be achieved. The ISTRESS project achieved this result thanks to the synergic improvement in the project knowledgebase of a) how process parameters change microstructure and stress state; b) how big is the range of tuning of residual stress gradient in the coating thickness; c) which microstructures and multilayer structures are beneficial to coating performance; d) the interplay between intrinsic stress fields and contact induced stress field in the resulting observed coating performance.

In the framework of the MS15 the Leoben University group tried another approach, focusing its coating design on toughness increase with two distinct multilayer strategies: in the first case the multilayer architecture was conventional, but the choice of the materials was made to maximally mismatch hardness or elastic modulus between contacting materials, keeping the other parameter well matched; in the second case the geometry of the interfaces was modified for the purpose. Mechanical tests performed on micro cantilever beam specimens of multilayered TiN/SiOx thin films (fig. 31) show that the fracture toughness of this hierarchical, microstructurally and mechanically heterogeneous material can be enhanced up to 60% with respect to either of its single layered constituents, which is attributed to a large difference in their...
with respect to either of its single-layered constituents, which is attributed to a large difference in their elastic modulus. Similarly, micro-bending tests of multilayered CrN/Cr thin films reveal an increase in fracture toughness of 40% with respect to CrN and Cr single layers. In this case, the enhancement of fracture toughness is attributed to the difference in strength of both constituents. For the second case study, toughness enhancement by interface geometry design, the Leoben group successfully deposited coatings with tilted brittle interfaces (fig. 31). The concept was applied to nanocrystalline monolithic TiN thin films which were mechanically tested as notched and un-notched micro cantilever specimens inside a scanning electron microscope. It was demonstrated that it is possible to increase fracture toughness of ceramic nanostructured materials of more than 150% by a dedicated grain boundary orientation design with respect to the direction of the expected crack path without loss of hardness. All these results were published in the Journal of Materials & Design and in Acta Materialia (section 4.2). Based on these results, the MS15 has been fully achieved as not only two coating algorithms were proposed to improve the coatings architecture, but also validated using number of demonstrators. The applicability of these tools were further extended to address a key industrial design problem and results show significant improvement in optimized coatings.

Stress data generation for advanced failure modelling of 3D IC (TSV, BEoL) and MEMS (MS16)
The milestone (MS) 16 deals with the delivery of an improved algorithm for MEMS and TSV design, based on the spatially resolved residual stress analysis. To achieve the objectives of this MS, in the first case a sample of freestanding MEMS and bi-layer TiW-Au cantilevers were produced at THALES and sent to Roma Tre for the analysis of their mechanical properties as well as residual stresses (fig. 32). The samples were characterized by an optical profilometer and scanning electron microscopy (SEM) equipped with energy dispersive x-ray spectroscopy (EDS), in order to acquire the information about the geometry, composition and the gap between the substrate underneath. The micro-beams were then deflected by using a specifically designed nanoindentation procedure based on dynamic stiffness measurement during bending, in order to extract the elastic modulus and the residual stresses of both layers. First, the classic beam theory was implemented for bilayer cantilevers enabling the extraction of elastic moduli. Then, residual stresses are estimated by deflecting double clamped beams, while implementing new analytical models for a bilayer system (fig. 32). The obtain elastic moduli are consistent with the average ones obtained for a single layer micro-cantilever and with nanoindentation results for TiW and Au homogeneous films. The residual stresses are in agreement with the values obtained from the double slot focused ion beam (FIB) and digital image correlation (DIC) procedure, providing an alternative and portable way for the assessment of residual stresses on composite double clamped micro-beams. The outcomes of this study was published in the Journal of Materials & Design (please see section 4.2 for more details).

For THALES, the results of this activity are of utmost importance, not only to address the process optimization but also the reliability issues of RF-MEMS devices. The nanoindentation experiments carried out at Roma Tre, which allowed the extraction of both Young’s Modulus and residual stress of each material constituting the RF-MEMS membranes had never been investigated before are very helpful to do retro-simulations in order to get a more accurate comprehension of the MEMS behavior and to simulate a design with optimized performances.

On the back of these new developments, a second set of samples with bilayer structures (TiW/Au) has been fabricated with the photolithography mask especially designed for the ISTRESS project and sent to Roma Tre for further analysis. The residual stresses have been effectively extracted for each layer implementing beam theory for this composite system. An elastic modulus of 70 GPa and 224 GPa have
Implementing beam theory for this composite system. An elastic modulus of 70 GPa and 224 GPa have been found for Au and TiW respectively on cantilevers which is in a good agreement with the monolayer value as well as with literature. Residual stress values of 270 MPa and 10 MPa was also achieved for TiW and Au respectively using the methods developed within the ISTRESS project.

In order to further extend the applicability of the methods, a collection of freestanding cantilevers and double clamped beams composed of tri-layer TiW-Au-TiW were produced by THALES and sent for analysis. The double clamped beams has a constant length of 120 µm, while the width varies between 5 to 20 µm. Once again, using the methods developed within the ISTRESS project, we were able to quantify both the elastic modulus (152.2 GPa) and the residual stress (142.5 MPa) in this configuration. It is interesting to note, that the elastic moduli of Au and TiW are equal to 79 and 260 GPa, respectively. The weighted average gives an elastic modulus equal to 161 GPa and it is in a very good agreement with the literature value. Further analysis on double clamped beams reveal that the elastic modulus values vary between 152 and 163 GPa for different width, however the average residual stress increases from 95.2 MPa (5 µm wide beam) to 162.2 MPa (20 µm wide beam) clearly indicating the increase in residual stress with the beam width.

The results achieved within the ISTRESS project especially on the multilayer structure are extremely useful for RF-MEMS activities at THALES. The configuration provides a good compromise in terms of actuation voltage (stiffness), switching time and gap according to the THALES specifications. Several mechanical cycling tests have been already performed on bi-layer and non-optimized tri-layer devices. Tri-layer systems exhibit a reliability of 108 cycles without any degradation of their global behavior @ 2 kHz with a duty cycle of 50%, while bi-layer structures show a loss of their properties (actuation voltage, insertion losses, isolation etc.) after 102 cycles under the same conditions.

It is possible that the results achieved within the ISTRESS project could lead to the fabrication of i) RF-MEMS that can handle high power (20W) without any auto-actuation and ii) stiffer membranes with shorter switching times due to the smaller gap and hence the production of faster devices.

All these results shows that that scientific advancements achieved within the ISTRESS project are extremely useful for industrial applications. It was for the first time that the reliable measurements of Young’s modulus of these bi-layers were achieved and clearly shows that the developed procedures are directly scalable to the MEMS industry quality control level and can be extremely important for supporting the design and optimization of novel micro-devices with improved performance not only RF-switches, but also accelerometers, pressure sensors and other kind of MEMS. On the basis of all these results, the milestone 16 was fully achieved.

Novel product design rules for micro/nanocrystalline materials for energy applications (MS17)

(i) Stress concentrators and cracks are associated with residual and 'live' stresses that vary on the micrometre length scale. Classical theories of deformation and structural integrity, such as Linear Elastic Fracture Mechanics (LEFM) and fatigue lifing approaches are based on mechanistic assumptions that historically stem from ‘thought experiments’, but in most cases lack direct experimental validation. For example, LEFM and Paris fatigue law assume that the Stress Intensity Factor (SIF) and its range provide a universal basis for establishing the correlation between the applied loading and the conditions for crack propagation. On the other hand, overwhelming evidence exists that these are limited, and fail to provide correct prediction in a number of important cases, when local deformation is strongly affected by such phenomena as grain-to-grain variation in stress and plastic strain, crack closure and crack blunting, crack branching, etc. In a series of studies, we made use of FIB-DIC in combination with other complementary techniques and modelling to reveal and quantify the influence of local residual stress and prior loading.
techniques and modelling to reveal and quantify the influence of local residual stress and prior loading history on fatigue crack growth rates (FCGR) (fig. 33).

(ii) Advanced polycrystalline alloys form the basis of modern energy technologies, from aeroengine design and power generation devices used on land and sea, to Li ion batteries for secondary energy storage. In a series of studies, we considered the effect of micron-scale residual stresses on structural integrity (fig. 34). Of particular interest to us were components made from high temperature resistant materials such as nickel-base super alloys by additive manufacturing. The challenge in this rapidly growing field concerns the ability to control the microstructure (grain orientation and morphology) and residual stresses at the grain level that exert crucial influence on the overall fatigue and creep resistance.

The capability of combining orientation mapping with stress calculation using anisotropic elasticity developed in this project opened up opportunities for intricate microstructure control by linking process control with subsequent residual stress mapping. In particular, we have been able to demonstrate that the build sequence exerts a prominent effect on residual stress evolution, and may lead to internal nano-voiding and cracking, unless precise control is exercised.

In D6.7 we focused our attention on the establishing and realising the capabilities of the new technique for the analysis of geometric and microstructural features at the scale and resolution attainable by the FIB-DIC ring-core method. To this end, we re-visited the well-established classification of residual stresses into the macro- and micro- types according to Macherauch. With the new technique in hand, we were in position to pose and resolve quantitatively the fundamental question regarding the correlation between the micro- and macro-scale residual stresses, since both of these are accessible by the FIB-DIC technique. We therefore undertook two case studies that we chose to serve as vehicles for our analysis. Firstly, we considered shot peened aluminium alloy samples with different geometry, in particular, with notches having root radii from 2mm and down to 0.15mm i.e. spanning over a decade of values. These polycrystalline samples were subjected to shot peening using identical conditions, but the residual stress states arising in them were different due to the distinct geometries. The stress profiles were determined using FIB-DIC, and it was possible to demonstrate that all cases can be described using common, consistent eigenstrain profile that acts as the most convenient means of process parametrisation. Some scatter was present: the results deviated from the general continuum prediction, although all results fell within a band around a master curve.

To investigate the nature of this phenomenon further, we performed FIB-DIC residual stress measurement on a four point bent beam aluminium alloy polycrystal sample. The overall macroscopic residual stress profile was predicted on the basis of an FE model that was carefully calibrated against the macroscopic stress-strain curve. EBSD was used to reveal the underlying grain structure. The statistical nature of the distribution of residual stress results around the macro-scale values was analysed, and it was demonstrated quantitatively that these values conform to a Gaussian distribution with the width of ~50 MPa. The origin of such variation is therefore not random measurement error, but rather the underlying variability of stress due to the elastic and plastic inhomogeneity that is associated with the very grain structure being studied.

We thus accomplished MS9 Effects of elastic-plastic inhomogeneities on stress relief and opened up an avenue towards a fundamentally new approach to structural integrity and durability analysis of materials and structures, based not merely on the statistics assumptions, but also backed up by specific numerical values of parameters. These advances fed directly into our contribution to MS17 Novel product design rules for micro/nanocrystalline materials for energy applications.

Stress analysis on polymers, polymer matrix composites and biomaterials (MS18)
Stress analysis on polymers, polymer-matrix composites and biomaterials (MS18)

The milestone (MS) 18 deals with the stress analysis on polymers, polymer matrix composite and biomaterials. Three case studies were carried out on different materials to achieve this milestone.

Case study # 1: The production of fixed partial dentures (FPDs) induces complex residual stress profiles, due to both the thermal expansion coefficient mismatch between the veneering ceramic and the framework and to the thermal gradients occurring during the final cooling. Detailed knowledge of residual stress distributions in the veneering ceramics is important to understand the interface phenomena with the framework and the consequences of the different firing systems. In this case study, residual stress distribution was analysed in heat-pressed ceramic on zirconia core with micrometre spatial resolution, with also a focus on the stress at the interface versus porcelain-fused-to-metal samples (fig. 35). The residual stress distribution was also correlated with the fracture toughness. Results revealed that both prosthetic systems showed a compressive stress at the ceramic surface on a micron-scale. Residual stress on a micron scale are higher in magnitude than the corresponding macro-scale values reported in the literature, due to the stress relaxation given, at larger scales, by micro-voids and cracks. The stress field was directly correlated with the indentation fracture toughness, which was higher in those areas where the compressive stresses were greater. Stress analysis in correspondence of interfacial porosity for the Zirconia sample also showed that micro-defects could induce local modifications of the residual stress field, which may even locally generate a tensile stress state.

Case study # 2: Human dental tissues have a hierarchical structure and versatile mechanical properties. The dentine enamel junction (DEJ) is an important biological interface that provides a durable bond between enamel and dentine that is a life-long success story: while intact and free from disease, this interface does not fail despite the harsh thermo-mechanical loading in the oral cavity. The underlying reasons for such remarkable strength and durability are still not fully clear from the structural and mechanical perspectives. One possibility is that, in an example of residual stress engineering, evolution has led to the formation of a layer of inelastic strain adjacent to the DEJ during odontogenesis (tooth formation). However, due to significant experimental and interpretational challenges, no meaningful quantification of residual stress in the vicinity of the DEJ at the appropriate spatial resolution has been reported to date. In this case study, we used the tools developed within the ISTRESS project for measuring the residual elastic strain at (sub)micron-scale. The reported results (fig. 36) span the transition from human dentine to enamel, and incorporate the material lying at and in the vicinity of the DEJ. The capability of observing the association between internal architecture and the residual elastic strain state at the micrometre scale is useful for understanding the remarkable performance of the DEJ and may help the creation of improved biomimetic materials for clinical and engineering applications.

Case study # 3: Carbon fiber (cf) reinforced polyether ether ketone (PEEK) composites have excellent tensile and flexural strength and a reduced elongation at break and impact strength at low temperature. Most common sources of residual stresses in these composite materials are from the discontinuities between the thermal expansion coefficients, yield stresses and rigidities or phase changes (e.g. cure shrinking) of different constituents. In this case study, residual strain analysis at (sub) micron-scale was carried out by utilizing focused ion beam (FIB) milling with digital image correlation (DIC) in both carbon fibers as well as PEEK matrix. The results (fig. 37) provide useful insight into the reinforced fiber that is in compression, while the matrix shows slight positive tensile stress. The detailed activity reporting on all these case studies is available in the ISTRESS deliverable D6.8. Based on these results, the milestone 18 was fully achieved.
Reference samples for method development and pre-standardisation (WP7)

The validation and error analysis of the FIB-DIC method was implemented in several Round Robin activities in order to identify and overcome actual limits of the method in terms of resolution and sensitivity with the aim to develop a robust standardised automated procedure for determination of average stress states and depth-resolved stress profiles in materials of interest. Round Robin tests on residually stressed materials were successfully performed on a series of representative reference samples prepared with specific microstructure and stress profiles on various substrates (typically silicon and steel) by physical vapour deposition. These included (i) a series of TiN samples exhibiting large stress gradients across the film thickness (C01, WD51) for validation of the sensitivity of the method in terms of depth-stress profile determination (ii) a series of TiN samples exhibiting either low compressive or low tensile stress without any pronounced variations across the film thickness (C02, WD52) for validation of the sensitivity of the method.

The data collected by the project partners were used to generate reference information to test and establish standardisation activities and were included in the Good Practice Guide providing guidelines for FIB milling and SEM image acquisition conditions with the aim of reducing the scatter and uncertainty in the measurements. The Round Robin tests were coordinated by NPL and the results were collected and described for sharing on a specifically developed online web 2.0 platform.

The following issues were addressed during Round Robin activities in three iterations:
(i) influence of the FIB milling parameters on the real shape of the trench and measured displacement field;
(ii) influence of SEM noise and SEM acquisition parameters on the measured displacement field after milling
(iii) optimization of the procedure for the achievement of sub-micrometre depth resolution for stress profiling

Furthermore, the collected data were successfully used for the development of design rules for advanced thin film and micro-device production within WP6.1 and WP6.2.

Potential Impact:
Impact as it was stated in the Description of Work

• The project is expected to realize a breakthrough in the measurement and modelling ability of the residual stress distribution at the (sub)micron-scale, thus proving industry with enabling technology for the design and production of innovative micro-devices with improved in-service performances, enhanced reliability and substantially reduced modelling costs.
• The achievement of a portable (i.e. scalable to industry) tool for the analysis of residual stress distributions with sub-micrometre resolution will enable industry to move from resource-intensive to real knowledge-intensive product development, with a relevant reduction of time-to-market and design/optimisation costs.
• New insights into the process-performance relationships of nano-layered systems, small-scale devices and amorphous materials, with a direct improvement of in-service life-time and reduced design/production costs.
• Foster significant advances in the field of sub-nanometre resolution displacement measurement from electron microscopy imaging, by developing innovative FIB milling and SEM imaging procedures and repeatable DIC routines.
• The project will quantitatively assess (through modelling and experiments) the ion-induced artefacts consequently to FIB milling, as a function of the ion dose and material properties.
consequently to FIB milling, as a function of the ion dose and material properties.
• This project will form the basis of the international standardization of the FIB-DIC methods for micron-scale residual stress analysis.
• Numerical modelling will be central to boost confidence in our understanding of material’s deformation response, the attended damage, possible artefacts, etc.
• The implementation of an automated digital image correlation (DIC) process through a graphical user interface (GUI) to retrieve the intrinsic stresses will greatly enhance the applicability of this method and lead to a better fundamental understanding in research as well as reducing development times in industry.
• The FIB-DIC GPG will incorporate most of the developments related to the measurement protocols and residual stress analyses for the range of materials of interest

Impact that is effectively demonstrated at the end of the project
• At the end of the project, a real technological breakthrough has been achieved in the specific field of micro/nano-scale residual stress assessment, as proved by developed FIB/DIC software (D8.7) validated FIB and DIC procedures (WP3 and WP5 deliverables) and the demonstrators with improved performance that were produced by THALES (D6.6) and BOSCH (D6.4).

• The examples reported in D6.6 (Thales) and D6.4 (Bosch) clearly demonstrate that a significant performance improvement can be achieved by design of optimal stress profiles, with NO significant increase of the manufacturing costs and a reduction of time-to-market.
• Deliverables D6.1 to D6.4 demonstrate that (a) residual stress distribution can have a dramatic effect on the mechanical behavior of layered systems and (b) real improvement of in-service life-time (wear and cavitation resistance) can be achieved by advanced (but not expensive) design of novel materials with tailored residual stress profiles. (BOSCH).
• Specific deliverables submitted to WP2 and WP3 demonstrate the achieved performance and validation of automated FIB procedures and DIC freeware. Such results will have absolute relevance for the nanomechanics community at large, where in-situ mechanical testing and strain analysis by DIC from SEM imaging is an increasingly investigated research field.
• Submitted deliverables within WP4 and WP5 (together with a significant number of published papers in international peer reviewed journal) show significant results and a first time in the literature for the experimental and simulation assessment of the ion-to-matter interaction mechanisms for the most relevant material classes.
• A series of Round-Robin activities (larger than the one anticipated in the DoW) was performed and reported within WP3 to standardization committees (VAMAS and ISO), establishing a solid basis to the future standardization of the method.
• A series of papers where published in international peer reviewed journals, reporting the quantitative assessment of (a) induced damage by the FIB gun, (b) effects of elastic anisotropy on strain relief and (c) effect of microstructural features on strain relief.
• DIC iSTRESS procedures has been published with detailed guidelines, and has already gained several thousand downloads.
• A good practice guideline is ready for publication and will be released by NPL shortly after the project. This document will become the reference worldwide for the application of the FIB-DIC method and will be a very solid basis for the standardization route of the method.

Impact for industry and SMEs in particular, as is was stated in the Description of Work:
• The innovative tools developed for the design of micro components will result in a significant reduction of
• The innovative tools developed for the design of micro-components will result in a significant reduction of the design costs of the components and a significant increase in their performance and life-time, with a corresponding increase in competitiveness of the developed product, with respect to non-European competitors.

• The ability to design optimized multi-layered nano-coatings with controlled residual stress profiles will enable industry (Bosch partner in this project) with relatively low-cost tools for product optimization (so, especially attractive for SMEs).

• The procedures developed in this project are expected to increase significantly life-time of micro-electronic devices, where the residual stress distributions are not normally considered in the design, and reduce the design costs, with a consequent relevant impact for SMEs.

• A cost analysis and optimization will be also performed within this project to establish the best strategies for test cost reduction, with a realistic final target of €1000/sample (which means €200/test) as the strategic threshold.

Impact for industry and SMEs in particular, what is effectively demonstrated at the end of the project:

• Several new design tools were developed during the project, as reported in deliverables D6.1 to D6.7 covering applications from thin film design, through micro-electronic device reliability assessment, to structural integrity analysis of polycrystalline alloys for energy applications. As demonstrated in D6.4 D6.6 and D6.7 results will lead to remarkably reduced designing costs for high-tech components in various industrial fields.

• In the specific case of Bosch, the iSTRESS project designed and produced TWO innovative demonstrators, which showed improved resistance to sliding wear and cavitation damage. Even more importantly, the produced multilayer coatings did not involve any additional production cost for the company, while the improvement in terms of performance is extremely significant.

• In the specific case of THALES, an RF-MEMS switch with an innovative three-layer structure was developed. The performance of the film was demonstrated to be superior with respect to single-layer structures. In addition, the project developed a novel FAST testing protocol, based on nanoindentation testing, which will probably allow for IN-LINE assessment of the residual stress and stiffness in MEMS devices.

• Roma TRE has already started some service activities for enterprises and SMEs. The cost at which the service is being sold is BELOW the anticipated threshold of 1000€/sample. Such a result poses FIB-DIC methods at the same level of and in direct competition with the more conventional low-resolution XRD approaches, whilst the cost for high-resolution synchrotron time is at least one order of magnitude higher.

The impact from the activities undertaken by the consortium in the course of the project has concerned establishing capabilities, disseminating good practice, making collaborative links, underpinning further development, and (finally and most important) pioneering the use of novel advanced methods by industries and SMEs.

The new micron-scale stress measurement capabilities, which were developed within the project on all major FIB/SEM systems, has been demonstrated through internal reporting, contributions to the Good Practice Guide, seminars and presentations at conferences, workshops, industry roadshows, and a very large number of high profile papers on international peer reviewed journals.

During iStress execution, we established a new paradigm of mechanical microscopy, the concept of obtaining spatially resolved information about such key material properties as elastic stiffnesses, hardness, plastic strain, residual stress, and fracture toughness. Another important aspect of impact has
hardness, plastic strain, residual stress, and fracture toughness. Another important aspect of impact has been the progressive incorporation of mechanical microscopy (micro-scale residual stress mapping) in the manufacturing and design practice at project full and associated partners.

In this context it is most important to mention the iStress industry partners and customers who have already experienced, at industry level, the use of the newly developed procedures:

1. Bosch (full partner)
2. Thales (full partner)
3. Rolls Royce (associate partner)
4. Saint Gobain (associate partner)

In case of Thales, the developed procedures will also be applied as a routine quality control tools, thus demonstrating the possibility of using such technique for in-line and real-time quality control.

Also in case of Saint-Gobain, the residual stress measurement is performed in between a fundamental processing step (tempering of the glass substrates) and can be considered as in-line testing.

The integration of the state-of-the-art nano-scale characterization tools into in-line and real-time quality control processes will have a dramatic impact on product development paths and thus on the quality and performance of specific products. This will contribute significantly to the enhancement of the competitiveness of European industry (especially in comparison with other emerging markets, e.g. China). The results achieved in the project allowed to establish general processing-structure-property models for multi-layered coatings with enhanced performance for specific applications. For such reasons, the impact of the developed technologies will not be restricted only to the specific fields of coatings e.g. for automotive industry, but can be readily expanded to other strategic sectors in the European industry, where improved fracture toughness in multilayer stacks presents a limitation to higher productivity, e.g. microelectronics, energy, textile.

The development of the technology and enhancement of the potential impact of this action will be further supported by the implementation of an Online Open Innovation Environment providing training and dissemination services that will be maintained after the project. Such an open web resource will have the main function of transferring to industry the advanced nanoscale modelling and characterization tools for optimization of nanostructured multilayer coatings.

The planned development of an Online Open Innovation Environment in the specific field of advanced residual stress measurement by FIB-DIC will dramatically improve the speed of material and/or nano-device development, bringing direct benefit in:

- Reduction in the time and resources required to find optima residual stress distribution giving rise to mechanical and functional performance;
- Innovative real time online/inline measurements for process control, optimisation and quality assurance, and improvement on current methodology used by industry where analysis of Residual Stress is possible by time consuming and destructive measurements only;
- Improving the accessibility of European raw materials, processing and manufacturing industry to high resolution materials characterisation infrastructures and advanced modelling tools.
- Development of stakeholders network sharing metadata in OIE through interaction with other European institutions, e.g. the European Materials Characterisation Council.

Specific impact for BOSCH
Specific impact for BOSCH
Diesel injection systems are subjected to complex load scenario over their life cycle, which includes large cycles (106 to 109) as well as high injection pressures (> 1400 bar during clean combustion). This is more pronounced with narrow gap widths where even a very small fraction would cause erosive and abrasive wear. Such wear will cause valve leakage, lowering fuel injection pressure and engine performance and ultimately increasing particulate emission from the engine. Particle sizes in the range of 5 to 20 µm are particularly critical. The figure below shows a typical injection system and its valve design with the seat cone inclined towards the ball axis along with the magnified view of the particles in the extreme load scenario. A common approach to protect against the complex load scenarios in sliding conditions is the coating of individual components, hence ensuring the prolonged function of these components over vehicle lifetime. The analytical simulations and optimization of protective coatings for particle damage using classical methods of sampling and testing is usually very resource intensive as not only each sample must be produced but also particle testing rigs are complex and costly to run and maintain. Furthermore, they do not permit to access the damage caused by individual particles for general conclusions but rather provide one defined point in a complex load scenario per test cycle. Therefore, a “single particle approach” can be more useful in accessing basic phenomena for defining design rules.

In the specific case of BOSCH, the global application of modern injection systems is enabled by robust design, also by the use of wear protective coatings. This prevents wear that causes valve leakage, lowers fuel injection pressure and engine performance, and increases particulate emission. During the ISTRESS project, a measurable improvement of the performance (adhesion) of coatings for diesel injection systems was achieved without increasing production costs, thanks to the improved design of multilayer coatings. In Europe, such a novel innovation approach is the main (and probably unique) way to compete with the extremely aggressively developing markets in the Far East.

As an example, more than 40% of all new cars are now equipped with a diesel engine due to the efficient fuel consumption. In the case of passenger cars, the main challenges to address concern high pressures (~ 2000 bar), low power consumption, light weight, low noise and long life time. Unfortunately, the varying quality of diesel fuel affects its ability to lubricate mechanical contacts between components, in some cases leading to component wear and reduction in the duration of reliable performance. The goal that was accomplished by the ISTRESS project is to address the process optimization to produce nanoscale multi-layer coatings to make a major impact in enhancing the performance of diesel engines, improving life cycle and reducing costs. This will open greater market opportunities for European industries through the superior quality and hi-tech product value. In addition, other industries such as thin film technology and micro-electronics will benefit from the results obtained in this project.

Specific impact for TESCAN
A methodology for stress estimation using FIB milling was developed and validated at TESCAN. Such method is still in a version that is not readily applicable at the production level and therefore is not attracting much interest among industrial end-users. We have, however, already seen some interest among our close collaborators that required local stress estimations at the micro-scale. This is illustrated in the following figure:

This interest is an indicator that the module would be useful for at least some industrial processes. However, the level of interest is quite low and it would take some considerable effort on the part of TESCAN (and other project partners that were involved in developing the method) to turn what is essentially a three-part workflow into a compact and easily applicable module. Following careful evaluation
essentially a three-part workflow into a compact and easily applicable module. Following careful evaluation of the required resource allocations, we will consider pursuing the development of this module into something that can potentially attract a wider customer interest. Until this development is complete, it is not possible to assess the full impact of the project results on our company.

In addition to this, as the results of this project as well as the possibilities of this method becomes more and more generally available (through the Good Practice Guidelines, papers and workshops), it is possible that there will be more of a demand for this method of stress estimation.

Specific impact for THALES

For Thales, Istress’ results are of utmost importance to address the process optimization and the reliability issues of RF-MEMS devices under development in the Group.

Nano-indentation experiments performed at Roma-Tre allowed extracting the values of the Young Modulus and the residual stress of each material constituting the RF-MEMS membranes. Those values that had never been investigated before are very helpful to do retro-simulations in order to get a more accurate comprehension of the MEMS behavior and to simulate a design with optimized performances. The RF-MEMS activity in TRT was based on multi-layer structures so far. Indeed, this configuration provides a good compromise in terms of actuation voltage (stiffness), switching time and gap according to Thales specifications.

However, the will of Thales to fabricate i) RF-MEMS that can handle high power (20W) without any auto-actuation ii) faster devices with a smaller gap inducing a shorter switching time, implies to get stiffer membranes.

Tri-layer membranes offer a good alternative to such conditions.

However, the residual stress depends of the anchoring of the membrane, the sacrificial layer (nature and thickness), the metallization of the membrane (nature of the materials and their thickness) and the deposition process. An experiment plan should help Thales to move towards a three-layer system with optimized stress by making a correlation between the reliability, the actuation voltage and the stress measured by nano-indentation at Roma Tre. This study is under investigation, a first set of tri-layer samples has been fabricated and has been delivered to Roma Tre.

Several mechanical cycling tests have been already performed on bi-layer and non-optimized tri-layer devices. Tri-layer systems exhibit a reliability of 108 cycles without any degradation of their global behavior @ 2 kHz with a duty cycle of 50%, while bi-layer structures show a loss of their properties (actuation voltage, insertion losses, isolation...) after 102 cycles under the same conditions.

Concerning Thales MEMS inertial/pressure sensors for Avionics purposes, numerous samples have been delivered during all ISTRESS project, preliminary with elementary layers, and finally with complete Thales Avionics MEMS pressure sensor structures.

In-situ DIC-FIB characterizations of MEMS Thales Avionics inertial/pressure sensors done during ISTRESS project have given a better knowledge of stress values into elementary layers, and stress distribution along sensor gages into complete MEMS pressure sensors.

MEMS pressure sensors simulations done during ISTRESS project (simulations without and with pressure load) have permitted a better understanding of failure mechanism modes under MEMS pressure load, with localization of regions sensitive to cracking. This work has also consolidated operating range for a better reliability.

These advances will bring new employees into relevant economy sectors, and improve the competitiveness of European manufacturing companies. As an example, TRT expect direct impact by transfer of methodologies to OTHER pilot lines within the company, e.g. for RF-MEMS devices, where
transfer of methodologies to OTHER pilot lines within the company, e.g. for RF-MEMS devices, where automated in-line mechanical testing of micro-membranes is also of paramount importance for product development.

Dissemination activities
Dissemination activities within WP8 consisted on a wide range of events and actions, with the main aim of bringing the news to the different communities (materials science, industry, metrology) of a novel method for residual stress analysis on a (sub)micron scale:
1. More than 45 papers have been published in international peer reviewed journal, with iSTRESS acknowledgment;
2. A special issue on “Advanced Residual Stress Analysis” was published in the journal “Materials and Design”, IF = 3.997;
3. More than 50 oral presentations were given at international conferences with iSTRESS acknowledgment;
4. A entire session of the 30th International Conference on Surface Modification Technologies (http://smt30.org) was dedicated to this project (D8.9);
5. An Industry road show was held in three different locations in Europe, to show the new FIB-DIC method to industry and SMEs in particular (D8.8)
6. Novel freeware for FIB and DIC procedures was released and is available online;
7. A Good Practice Guideline on the FIB-DIC methodology for residual stress analysis has been prepared and will be published soon after the project;
8. Project liaisons with CEN and ISO standardisation committees have been established and will be maintained after the project to establish a standardisation path for the project
9. Finally, the publication of an online Open Innovation Environment, which will be maintained well beyond the end of the project, where registered users will be allowed to share their data, to find online tutorials on the technique and to have open discussions with experts in the fields and with enterprises (including SMEs) and other stakeholders

Wider dissemination through the organization of a conference session and an Industry Road Show
The milestone (MS) 25 deals with the wider dissemination activities of the ISTRESS project including peer reviewed publications, webinars and presentations at workshops and conferences.
The peer reviewed publications were continued throughout the course of project and more than 40 publications were made on various activities of ISTRESS project in high impact factor journals such as Nano energy (IF: 11.553) Acta Biomaterialia (IF: 6.008) Acta Materialia (IF: 5.058) Journal of Materials & Design (IF: 3.997) Scripta Materialia (IF: 3.305) etc.
A dedicated special issue namely “Residual stress evaluation at the micro and nano scales: recent advancements of measurement techniques, validation through modelling and future challenges” was also published in the journal of Materials & Design. The purpose of this special issue was the distribution of results to a wider scientific community using a dedicated platform. Publications in such quality journals is a clear indicator that the activities carried out within the ISTRESS project yield excellent results and are of high scientific value. On the back of these high quality publications, it is expected that both the scientific community as well as the industry will be benefited from the results achieved within the ISTRESS project.
A complete list of all peer reviewed publications and oral presentations at conferences is available in section 4.2 of this document.
As part of dissemination activities, presentations related to ISTRESS activities were carried out at the 30th international conference on Surface Modification Technologies (SMT30) at Politecnico di Milano, Milan.
International conference on Surface Modification Technologies (SMT30) at Politecnico di Milano, Milan (Italy) from 29th June – 1st July, 2016. The conference was aimed at fostering research on surface engineering, promoting related international cooperation among scientists and engineers and providing means for the public dissemination of the results from these efforts. The conference interest includes both industrial materials, e.g. metals, alloys, polymers, ceramics, composites, and advanced materials and processes under development or used in particular applications. The conference brings together users, producers and researchers, both engineers and scientists who have a common interest in surface materials behaviour.

At the SMT30, 18 oral presentations were made by the partners covering specifically mechanical properties of engineering materials and residual stress measurement in wide range of materials. The presentations also cover the industrial aspects in detail and number of key design problems were discussed. More details about the conference activities along with the list of talks were reported in ISTRESS deliverable D8.9.

Included in the project tasks is a “road-show” idea where there will be demonstrations in three different member states for industrial users. Project partners agreed that it will be the responsibility of TESCAN where the participants will be able to see for themselves what can be done using a FIB-SEM for measuring relative stress.

Three workshops were organized in three different member states – Italy, France and the United Kingdom. In two cases, the workshops were accompanying events at prestigious international conferences and the final one was held at the instrument placed at one of the project partners who contributed to the development of the stress-measurement workflow.

The schedule of the workshops were as follows:
- The 30th International Conference on Surface Modification Technologies, 29th June – 1st July 2016, Milan, IT
- The European Microscopy Congress 2016, 28th August – 2nd September 2016, Lyon, FR
- 15th of November, 2016 - MBLEM, ETB Building, Department of Engineering Science, University of Oxford, UK

In each workshop a TESCAN application engineer demonstrated the three-part workflow involving automatic FIB milling and image acquisition, DIC analysis and strain to stress conversion using a curve fitting function.

Information about these workshops was widely disseminated before the event. During the conferences, wide publicity was given to draw the attention of interested participants during the lecture sessions. The number of participants that attended these workshops indicates that the possibility of using FIB milling to estimate residual stresses is still not widely known and this is a challenge for the future to spread awareness about this method among potential users.

Drafting and publication of a good practice guideline (MS22)

The FIB-DIC Good Practice Guide (GPG) is one of the key outputs from the iSTRESS project, aimed at providing users with practical advice for making reliable residual stress measurements on their own systems and materials using this technique. It brings together the expertise and experience of the project partners in a single document, and will be used to promote the transfer and uptake of the newly developed methodologies to a wider industry. Led by NPL and Uniroma3, it is based largely on input and technical expertise of the project partners involved in WPs 2-5, and the results and findings from the various round robin exercises and studies carried out during the project. The GPG covers all aspects of the measurement process and residual stress analyses for the range of materials and sample geometries.
Measurement process and residual stress analyses for the range of materials and sample geometries examined within the iSTRESS project. It is designed to be a practical guide, with advice, supporting information to illustrate the various process steps and data analysis required to make reliable and repeatable high resolution residual stress measurements. Individual sections of the guide cover recommendations for imaging, milling, DIC analysis, modelling and data analyses, as detailed below.

The Structure of the Guide is:

• Section 1: Scope
• Section 2: Definitions and symbols
• Section 3: Introduction
• Section 4: Development of the FIB-DIC approach
• Section 5: The FIB-SEM
• Section 6: Sample issues
• Section 7: Surface patterning
• Section 8: Milling conditions and issues
• Section 9: Recommended FIB milling geometries
• Section 10: SEM imaging
• Section 11: Digital Image Correlation
• Section 12: FE Modelling
• Section 13: Data analysis
• Section 14: Correcting for FIB induced Eigenstrain
• Section 15: Validation
• Section 16: Errors and uncertainty
• Section 17: Reporting of results
• Section 18: References
• Appendix 1: General procedure for FIB-DIC milling
• Appendix 2: Influence functions for residual stress analysis
• Appendix 3: iSTRESS software for DIC and stress calculation

The GPG will be published as a standalone document (NPL GPG143) and will also be available on the iSTRESS project. The GPG forms an important document for pre-standardisation of the FIB-DIC technique and will continue to be promoted to the Standards community through CEN TC/352 and VAMAS TWA22. It is expected to form the basis of a future International standard in this field.

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