Publishable Summary for 14IND03 Strength-ABLE
Metrology for length-scale engineering of materials

Overview

Conventional understanding of the strength of materials, states that a large beam and a tiny beam of the same material will fail at the same stress. However, in the real world, small beams/fibres are stronger and this ‘size-effect’ or ‘length-scale effect’ can change the strength of a material by an order of magnitude. This project generates design rules and new measurement techniques to exploit the opportunity to length-scale engineer materials into more sustainable/energy efficient industrial components that are lighter, stronger, fatigue and wear resistant.

Need

The strength of a material is a key design parameter and needs to be known for the design of all manufactured components. For metals and ceramics, material strength is not only determined by intrinsic microstructural length scales, but also the extrinsic dimensions of the material component especially when they are made small; in addition, the measurement method also introduces a length scale. All these length scales play a role in determining the measured mechanical response, in this case the strength. However, the conventional assumption underlying Finite Element Analysis (FEA) models is that the yield stress is constant across all length-scales; they are therefore unable to determine the very real material performance improvements available through length-scale engineering. There is a need for better understanding of how different length-scales combine to give overall strength, both at ambient and elevated temperatures. This need is increasingly urgent as nano-technology is opening up opportunities for the production of materials with large reductions in microstructural length-scales and correspondingly introducing major possibilities for major strength increases. The material strength requirements for light-weighting to reduce energy use and CO_2 emission in both transport and industry could be met by better exploiting these length scale effects to make stronger/lighter components. Even pure metals can be made extremely strong by length-scale engineering their characteristic dimension to the nano-scale, which could be used to remove the need for expensive or toxic additives to alloys previously used to enhance strength, and could also open up new sustainable low-energy manufacturing routes. One such route is electrochemical additive manufacturing, which can deposit pure metals and simple alloys as net shape components with a nanoscale structure that would make them super-strong.

Objectives

The overall aim of the project is to measure and understand plasticity size effects, to meet the measurement challenges that size effects pose, and to realise the material and component performance benefits that length-scale engineering offers. Indentation and uniaxial compression testing methodologies are included as well as contributions to standardisation bodies.

The specific technical objectives of the project are to:

1. **Develop validated design rules for combining different size effects** to optimise strength and toughness of materials and components over a range of temperatures. Develop a plasticity size effect algorithm for processing small scale test data maps to obtain material-only properties. This algorithm will be incorporated into an analysis programme to support materials property mapping (by indentation) of surfaces and small volumes of materials. The project will enable the combination of multiple length-scale effects and reduce the uncertainty in property and performance prediction by an order of magnitude (from factors of 2 or more to tens of percent).
2. **Improve nano- to micro-scale property measurements** by developing a MEMS-based instrumented indentation (IIT) system to bridge the length and force-scale (<1µm to ~10 µm, 1 µN to ~few hundred

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μN) between AFM and nanoindentation; developing better-defined-shape probes with lower uncertainty contact sizes; and improving AFM-based property measurements. The project will develop tips down to less than 10 nm in size with ~1 nm precision, and/or with high aspect ratio, exact shape and high repeatability. These tips will also be stronger (up to 100 GPa), cheaper (~1/10th of cost), better-defined-shape that will reduce the key uncertainty of tip contact area from ~50 % to ~10 %.

3. **Develop methods and associated uncertainty budgets to determine the length-scale dependence of the strength and toughness of materials vs. temperature.** The methods will have sufficient resolution to distinguish between model predictions and will use:
    - Characterisation methods, such as for dimension of a particle, structure or layer, grain size, crystal orientation and crystal rotation, residual stress.
    - High-resolution indentation and compression (vs. temperature) of surfaces, micro /nanostructures.
    - Systematic quantitative variation or estimation of dislocation density.

    The project will establish a new NMI capability in high temperature indentation, at temperatures up to 400 °C in air and at least 500 °C *in vacuo* with a reduction of uncertainties to 10 % in high T indentation modulus and hardness. In addition, the project will establish a new capability for high temperature, high spatial resolution (lateral <10 µm, depth ~10 nm), indentation creep measurement.

4. **Develop and evaluate new measurement methods** to distinguish between the contribution(s) to the total test response from test-related size effects (i.e. Indentation Size Effect) and that from size-related strength or plastic deformation properties of a particle, volume, or structure. In addition to conduct a feasibility study to determine the capability of the measurement methods to characterise and map difficult to measure properties such as material dislocation density and mobility, stacking fault energy and plastic deformation zone size.

5. **Support the competitiveness of EU industry** by engaging with industries using manufacturing technologies and process control and by supporting the development of new, innovative products. This includes conducting case studies to facilitate the uptake of the technology and measurement infrastructure developed by the project; and by contributing to standardisation bodies.

Progress beyond the state of the art

This project focuses on the traceable measurement and characterisation of size effects in the properties of materials so that they can be exploited in improved measurement methods which will lead to the design and manufacture of new or improved industrial components. The project has generated outputs that extend instrumented indentation beyond state of the art as well as the understanding and modelling of the plastic deformation of materials that deform via dislocation motion. A novel finite element package has been developed incorporating length scale effects. Validation of this new model has now been carried out and it shows good agreement with experiments. New diamond probes have been developed with very well defined tip geometries which have been combined with a new MEMS based micro mechanical test system to provide improved methods of testing which will extend the range of traceable measurement methods to lower loads than achievable by conventional nanoindentation. These MEMS based micromechanical test systems have been verified and validated by tests carried out by partners. This project has developed the elevated temperature indentation in IND13 T3D to higher temperatures and *in vacuo* to enable a wider range of materials and test conditions to be explored. This includes the purchase of a new test system that has now been validated and verified which is fitted with a vacuum system and with gas inlets options to provide a solution to reduce surface oxidation which is an important issue for surface mechanical study. Testing with this new test system have been carried out at temperatures up to 500 °C on copper. New high temperature micro-mechanical test procedures have also been developed which show promise for the determination of high temperature materials properties. The current project has carried out an extensive programme of work to determine the length-scale dependence of plastic deformation of materials that deform via dislocation motion. A comprehensive testing matrix has been carried out across the length scales on a range of appropriate materials which is currently being evaluated to define the experimental basis for length scale engineering.

Results

Develop validated design rules for combining different size effects

The current state of the art plasticity size effect theories were reviewed in the early stages of the project. An analytical model has been developed and applied to enable combining different size effects (such as indentation size effect, grain size effect, dislocation work-hardening). This analytical model has been used as the basis for new finite element code that has been validated against experiment. In addition, a crystal plasticity finite element model, which also supports discrete dislocation dynamics modelling, has been developed to implement the proposed theory to simulate the combined plasticity size effects. These simulations have been used to develop deep understanding of the plastic deformation mechanism at small scales to support the development of the simplified analytical model as a useful tool to predict the strength material system of interests. The aim is to develop validated design rules that could be easily adapted in industry to achieve stronger component with reduced cost. These modelling approaches have been validated by comparison with experimental results and have been shown to fit experiment reasonably well.

Improve nano- to micro-scale property measurements

A MEMS nanoindentation system using a compact 3D micro- and nano-positioning system for sample engagement has been built. The MEMS actuator which was developed in the University of Chemnitz has been combined with a new test system developed at PTB to produce a working test system. Validation trials have taken place which have shown that the system operates well and produces useful results. The operating envelope of the new instrument has been established. The new MEMS based test system has been made available to members of the project consortium who have tested the system; opprtunities for the commercialisation of this test system whave been pursued. A 3rd generation of single crystal diamond AFM probes for MEMS nano-indentation has been produced which have further improvements to their geometry and even higher spring constants. The functionality of using these probes in an AFM system has been demonstrated. These test probes show many advantages for low load mechanical testing over the traditional silicon styli.

Develop methods and associated uncertainty budgets to determine the length-scale dependence of the strength and toughness of materials vs. temperature.

A considerable volume of work has been carried out on characterising materials. Many of these materials have subjected to significant programmes of mechanical testing across different length scales. This mechanical testing has now been completed. A specific example of this work is high temperature in situ testing by EMPA where a number of different approaches have been adopted to evaluate the time dependent deformation behaviour of the materials. A specific aspect of the mechanical testing exercise has been carried out on appropriate alloys at UKAEA. INRIM has carried out macroscale hardness measurements and uniaxial tensile tests. The installation of new NMI measurement capability at NPL in high temperature indentation testing which has now been completed and verified according to the appropriate international standards. Experiments have been carried out on nanoindentation of copper at temperatures up to 500 ^{o}C. The instrument has the capability to carry out measurements at temperatures up to 900°C in high vacuum.

Develop and evaluate new measurement methods

A number of different test methods have been developed which give new information on the length scale dependence of materials. These include the use of spherical indentation, mapping of length stress strain measurements, and macro-scale instrumented indentation. The analysis of data acquired in other work packages with the new analysis models that have been developed confirms that successful measurement of material properties including length scale effects can be carried out. An example of this is the large programme of measurements, modelling and analysis that were carried out on CuCrZr alloys where the material response was described well over several length scales.

Support the competitiveness of EU industry

Industrial partners participating in this project have provided materials according to their industrial requirement. The consortium is also actively in contact with relevant industry and encourage the important stakeholders to become collaborators. An early success in a case study has been the delivery of a prototype length-scale enabled injection manifold that promised major improvements in functionality. A New ISO standard (ISO14577 Part 7) for Instrumented indentation test at elevated temperature is being developed.

Impact

Dissemination of the results of the project was carried out by a number of different routes. By the end of the project the following targets were achieved: 3 case studies, stakeholder meetings, 30 peer reviewed papers, 36 conference presentations and posters), 10 industrial visits, input of project results into standardisation, 2 press releases and 2 trade articles, 13 training activities availability of new mechanical test systems and new analysis software.

Impact on industrial and other user communities

There has been some industrial impact through the case studies being developed under objective 5. Thus in one case study there has been delivery of a prototype length-scale enabled injection manifold that is promising major improvements in functionality. Industrial interest in the project is shown through the addition of eight additional collaborators and stakeholders to the project, including Diehl Metal Application GmbH, Italian Aerospace Research Center (CIRA), Maschinenfabrik Kaspar WALTER GmbH & Co.KG and Bruker nano GmbH. Interest in the project has also been shown by GETec who are interested in applying the technology developed in the project to their AFM/SEM product.

Impact on the metrology and scientific communities

The project has produced improved interpretation of mechanical test responses from small samples and small-scale test methods through the analysis systems being developed in response to objective 1. In addition, in response to objectives 2 and 3, new test methods using size effect responses to measure, non-destructively, material length-scales and intrinsic properties have been generated, providing new tools for characterisation and investigation of materials. In particular, interest from potential users has been shown in in the new diamond probes with improved tip geometries that have been developed, and also in the development of the new MEMS based low load indentation test system that has now been validated and verified.

In response to the requirement in objective 5 for knowledge transfer, there have now been thirty publications from the project and 36 conference presentations have been made. A well-received workshop on the application of length scale engineering was also held in September 2017 at the University of Chemnitz which presented the results of the project to participants from technical institutes from the area. Several other training events have taken place at industrial organisations.

Impact on relevant standards

As part of the work to respond to objective 5, the online training mentioned in the previous section will be fed into the BIPM Consultative Committee on Mass and its Working Group on Hardness, and into ISO/TC 164/SC3 on Hardness. Input of output from the project into the work of ISO/TC164/SC3 on the interpretation and comparison of hardness results at different length-scales is continuing with attendance by project stakeholders at a meeting in September 2017. In particular, a new work item is being drafted to provide guidelines for instrumented indentation testing at elevated temperature: ISO 14577-part 7: Instrumented indentation test at elevated temperature.

Longer-term economic, social and environmental impacts

The research carried out in this project has generated a range of improved measurement methods and a more unified understanding that enables significantly improved component performance/lifetime with consequent operational and environmental cost reductions. The outputs of the project have been tailored to be of interest to industry in order to attract stakeholders, and include:

- New models, algorithms, software and design rules for plasticity size effects and hardness calibration and testing
- Nano to micro scale property measurements at room temperature and elevated temperature
- Better indentation probes and a MEMS-based instrumented indentation system
- Non-destructive and potentially *in situ* methods for obtaining stress-strain curves by indentation e.g. for quality control or structural health monitoring.
- Industrial case studies to demonstrate effectiveness to a range of industrial sectors, such as demonstrated new innovative length-scale engineered components and new low-energy additive manufacturing routes.

- Training material for length-scale engineering for strength aimed at industry and design engineers

List of publications

[1] Michler, J., Wehrs, J., Mohanty, G., Di Gioacchino, F., Edwards, T.E.J. and Clegg, W.J, .*Longitudinal twinning in a TiAl alloy at high temperature by in situ microcompression, Acta Materialia*,148 (2018) 202-215, https://doi.org/10.1016/j.actamat.2018.01.007

[2] Tarleton, E., Hofmann, F. and Das, S., Consistent determination of geometrically necessary dislocation density from simulations and experiments, International Journal of Plasticity, 109 (2018) 18-42, 10.1016/j.ijplas.2018.05.001

[3] Coppola, G., Cagliefo, R., Genta, G., Maizza, G., Barbato, G., Germak, A., Origlia, C. and Schiavi, A.Measurement of macro-scale indentation modulus using the primary hardness standard machines at INRIM, IMEKO (2017), https://www.imeko.org/publications/tc5-2017/IMEKO-TC5-2017-001.pdf

[4] Jennett, N. M., and Hou, X. D., A method to separate and quantify the effects of indentation size, residual stress and plastic damage when mapping properties using instrumented indentation, Journal of Physics D: Applied Physics, 50 (2017), 45, https://pure.coventry.ac.uk/ws/portalfiles/portal/12629973/Hou_and_Jennett_Accpeted_version.pdf

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[6] Hainsworth, S.V., Kareer, A., Hou, X.D. and Jennett, N.M., The interaction between Lateral size effect and grain size when scratching polycrystalline copper using a Berkovich indenter, Philosophical Magazine 96 (2016) 32-34, https://pureportal.coventry.ac.uk/files/12969492/The_interaction_between_Lateral_size_effect_and_grain_size.pdf

[7] Hainsworth, S.V., Kareer, A., Hou, X.D. and Jennett, N.M., The existence of a lateral size effect and the relationship between indentation and scratch hardness in copper, https://pure.coventry.ac.uk/ws/portalfiles/portal/10344470/Nigel_Jennett_Manuscript_revised.pdf

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<tr><td colspan="2">Project start date and duration:</td><td>1st June 2015, 3 years</td></tr>
<tr><td colspan="3">Coordinator: Mark Gee, NPL Tel: +44 20 8943 6374 E-mail: mark.gee@npl.co.uk
Project website address: http://empir.npl.co.uk/strength-able/</td></tr>
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Partner 1 NPL, United Kingdom
Partner 2 BAM, Germany
Partner 3 CMI, Czech Republic
Partner 4 DFM, Denmark
Partner 5 INRIM, Italy</td><td>External Funded Partners:
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Partner 7 QMUL, United Kingdom
Partner 8 TU-Ch, Germany
Partner 9 UCov, United Kingdom
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