Integrated experimental-numerical methodology to map microstructural strain and stress evolution in bulk nanostructured alloys

C.C. Tasan, D. Yan, M. Diehl, F. Roters P. Shanthraj, C. Zambaldi, D. Raabe

Max-Planck-Institut für Eisenforschung,
Max-Planck-Straße 1, 40237 Düsseldorf, Germany

Microscopic strain and stress partitioning among the present phases govern the macroscopic mechanical response of multi-phase alloys. However, these phenomena are challenging to probe due to the multiple scales involved, dynamic nature of the deformation process, and lack of dedicated methodologies. The challenge is even more enhanced for multi-phase alloys with features smaller than micrometer scale.

A novel approach is introduced in this work that strengthens the connection between microstructure and mechanical properties in such alloys. To this end, numerical simulations and experiment analyses are carried out in an integrated manner to investigate the microstructural deformation mechanisms in multiphase microstructures.

For the experiments, a novel, *in-situ* SEM imaging and digital image correlation (DIC) based methodology is developed that allows concurrent microstructure and strain mapping at sub-micron resolution. Strain mapping is achieved by the application of a layer of nanoparticles on the sample surface (as the DIC pattern) that are selectively imaged during deformation. Concurrent microstructure mapping is achieved by optimization of imaging conditions to minimize the interference of the DIC pattern nanoparticles.

For the simulations, the electron backscatter diffraction (EBSD) map of the undeformed state of the same in-situ investigated microstructural patch is used to create the model. 2D full-field crystal plasticity simulations are run employing a recently developed spectral solver suitable for high-phase contrast materials [1, 2] implemented in the crystal plasticity simulation-kit DAMASK [3, 4]. The individual phase properties are obtained by additional inverse CP simulations of nanoindentation experiments carried out on the original microstructure [5].

The methodology will be demonstrated here on the example of martensite-ferrite dual phase (DP) steel, for which promising correlation between the simulations and experiments is achieved, despite the complex micro-mechanics of this material.

[1] P. Eisenlohr, M. Diehl, R.A. Lebensohn, F. Roters: Int. J. Plasticity 46 (2013), 37 - 53
[2] P. Shanthraj, P. Eisenlohr, M. Diehl, F. Roters: Int. J. Plasticity (2014) submitted
[3] http://damask.mpie.de
[4] see talk by F. Roters in Symposium E
[5] C. Zambaldi, Y. Yang, T. R. Bieler, D. Raabe: Journal of Materials Research 27 (2012), 356-367

Microstructure-sensitive modeling of the mechanical behavior of polycrystalline materials with direct input from emerging 3-D characterization methods

Ricardo Lebensohn, Reeju Pokharel

Los Alamos National Laboratory, Los Alamos, NM 87545, USA

Emerging characterization methods in experimental mechanics pose a challenge to modelers to devise efficient formulations to permit exploitation of the massive amount of data generated by these novel techniques. In recent years, we have developed Fast Fourier Transform (FFT) based methods for polycrystals (e.g. [1]), which can efficiently use voxelized microstructural images of heterogeneous materials as input to predict their micromechanical and effective response.

Furthermore, models based on polycrystal plasticity are increasingly used in engineering applications. These models require a proper consideration of the single crystal plastic deformation mechanisms, a representative description of the microstructure, and an appropriate scheme to connect the microstates with the macro response. FFT-based methods are ideal candidates to fulfill these requirements, albeit at the expense of numerically very demanding computations.

In this talk, we will discuss the latest numerical implementations of the FFT-based formulation for complex constitutive behaviors [2,3] and amenable to input and validation from 3-D characterization methods [4], as well as our recent efforts to embed this kind of models in Finite Elements, to solve complex boundary-value problems with microstructure-sensitive material response.

[1] R.A. Lebensohn, A.D. Rollett and P. Suquet: “Fast Fourier Transform-based modelling for the determination of micromechanical fields in polycrystals”. JOM **63**, 13 (2011).
[2] R.A Lebensohn, A.K. Kanjarla and P. Eisenlohr: "An elasto-viscoplastic formulation based on fast Fourier transforms for the prediction of micromechanical fields in polycrystalline materials". Int. J. Plast. **32-33**, 59 (2012).
[3] R.A. Lebensohn et al.: "Modelling void growth in polycrystalline materials", Acta Mater. **61**, 6918 (2013).
[4] R. Pokharel et al.: “Polycrystal plasticity: comparison between grain scale observations of deformation and simulations”, Annu. Rev. Condens. Matter Phys., in press.

Computational Process-Structure-Properties Modeling of Thermal Sprayed Coatings

Tatu Pinomaa, Anssi Laukkanen, Kenneth Holmberg, Erja Turunen, Tarja Laitinen

VTT Technical Research Centre of Finland, Espoo, Finland

We present a predictive multiscale model for microstructure formation and evaluation of material properties in thermal spray coatings. The model is linked to a multiscale nano-microstructure based continuous material model to predict material properties and wear resistance of powder metallurgy based end products.

The studied coating technique is called high velocity oxygen-fuel spray (HVOF). In this process, oxygen and fuel is ignited inside a nozzle, leading to a high temperature exhaust gas flow that – due to the high combustion pressure – accelerates to supersonic velocities. Coating powder is injected into this gas stream, which is dragged into high velocity, and is partially or fully molten in flight. The powder particles hit the surface, and during several microseconds, simultaneously deform into disk-shaped "splats". Subsequent particle impacts lead to a lamellar coating microstructure. Based on specified process parameters, we use computational fluid dynamics (CFD) to describe a reacting and supersonic gas flow inside the nozzle, followed by a lagrangian particle model for the propagation and heating of injected coating particles. This model provides particle velocities and temperatures upon impact, which are used as input for a phase field simulation of coating particle impact and rapid solidification. Subsequent particle impact simulations can be used to model the coating formation.

This process-structure model, complemented with metallurgical and thermodynamical information yields the microstructure for a structure-property computational model. The process analysis results are coarse grained to yield a statistically representative model of the thermal spray coating, the structure being explicitly included in the imaging based property computation utilizing a finite element approach. The nano-microstructural model is subjected to simulated testing for its mechanical and wear responses, and a feedback loop to the coating processing model is presented for material tailoring. The microstructure can be linked to a multiscale representation of material response on larger spatial scales using methods such as the Arlequin method, which provide the basic material properties of the coating in a product and component environment, e.g. porosity, splat-substrate adhesion, and splat-splat cohesion and their effects can be evaluated and quantified. The methodology can also be used to link the coating response to specific external environments – for example degradation due to wear, high temperatures, or erosion-corrosion.

An inverse optimization strategy to determine single crystal mechanical behavior from polycrystal tests: application to Mg alloys

V. Herrera-Solaz[1], Javier Segurado[1,2], Javier LLorca[1,2]

[1]Department of Materials Science, Polytechnic University of Madrid, 28040 Madrid, Spain
[2]IMDEA Materials Institute, 28906 Getafe, Madrid, Spain

An inverse optimization strategy was developed to determine the single crystal properties from experimental results of the mechanical behaviour of polycrystals. The optimization method is based on computational homogenization of the polycrystal behavior by means of crystal plasticity finite element simulations of a representative volume element of the microstructure in which the dominant slip and twinning systems were included in the constitutive equation of each grain [1-2]. The inverse problem was solved by means of the Levenberg-Marquardt method, which provided an excellent fit to the experimental results. The iterative optimization process followed a hierarchical scheme in which simple representative volume elements were initially used and successively by more realistic ones to reach the final optimum solution, leading to important reductions in computer time.

The new strategy was applied to identify the initial and saturation critical resolved shear stresses and the hardening modulus of the active slip systems and extension twinning in Mg alloys. It was found that if only two stress-strain curves were used as input in the optimization procedure, the parameters obtained from optimization were not able to accurately predict the mechanical behaviour of the polycrystal under different loading conditions. Thus, a minimum of three independent stress -strain curves was necessary to determine the single crystal behaviour from polycrystal tests in the case of highly textured Mg alloys.

This methodology was applied to study the critical resolved shear stresses (CRSS) of two magnesium alloys, MN10 and MN11 containing 0.5wt.% and 1wt.% Nd, respectively. The initial CRSSs were observed to change drastically with increasing RE content. In particular, the CRSSs of basal and twinning modes increased while the CRSSs of pyramidal and prismatic systems decreased to an extent that all values become similar for alloying additions of 1wt.%. This is consistent with the isotropic yielding behaviour observed in the MN11 alloy. The changes in CRSS with RE addition lead to the promotion of twinning at the expense of basal slip and to enhance the activity of non-basal systems.

[1] J. Segurado, J. Llorca. Simulation of the deformation of polycrystalline nanostructured Ti by computational homogenization. *Computational Materials Science*, **76**, 3-11, 2013.
[2] V. Herrera-Solaz, J. LLorca, E. Dogan, I. Karaman, J. Segurado. An inverse optimization strategy to determine single crystal mechanical behavior from polycrystal tests: application to AZ31 Mg alloy. *International Journal of Plasticity.* In press.
[3] V. Herrera-Solaz, P. Hidalgo-Manrique, M. T. Pérez-Prado, D. Letzig, J. LLorca, J. Segurado. Effect of rare earth additions on the critical resolved shear stresses of magnesium alloy. Submitted for publication.

Identification of constitutive parameters by inverse simulation of indentation in single crystals and close to grain boundaries

Claudio Zambaldi[1], David Mercier[1], Yang Su[2], Philip Eisenlohr[2], Thomas R. Bieler[2], Martin A. Crimp[2], Dierk Raabe[1], Franz Roters[1]

[1]Max-Planck-Institut für Eisenforschung, Düsseldorf, Germany
[2]Michigan State University, East Lansing, MI, USA

We present findings based on a recently introduced method [1,2] to extract critical resolved shear stresses and hardening parameters from quasi single crystal indentation. Individual grains of known orientation were probed by nano- or microindentation far away from the grain boundaries for their single crystal mechanical response. The resulting pile-up topographies were characterized by atomic force microscopy or confocal microscopy. In this way the orientation dependent activation of different deformation systems was analyzed by comparing the measured pile-up topographies against crystal plasticity finite element simulations of the indentation process.

Non-linear optimization of the constitutive parameters for the underlying crystal plasticity model, allowed us to extract a single set of parameters which represents the experimentally observed data for all indented orientations. In this way the relative activation of plastic deformation modes could be quantified based on simple indentation experiments.

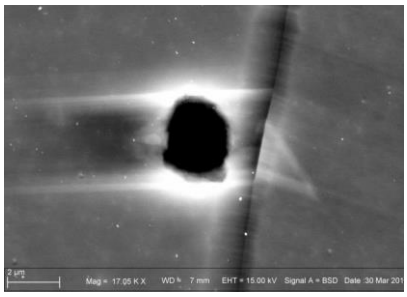

Figure 1: Nanoindentation close to a grain boundary in magnesium. Twinning is initiated in the adjacent grain.

After the initial development of this method on pure titanium [1] it has been applied to a number of metals and alloys with hexagonal (titanium alloy, magnesium [3]) and bcc (ferrite phase [4], tantalum, molybdenum) structure. The specific findings for the relative activation of different mechanisms in each phase will be presented and discuss.

Furthermore, the approach to quantify micromechanical effects by evaluating the anisotropic pile-up profile was extended to characterize the micromechanical effect of grain boundaries. In this case the possibility to efficiently probe many locations on a sample was important since it enabled the rapid acquisition of experimental data. Again the topographic data was compared to crystal plasticity finite element simulations of the deformation during indentation of a bicrystal. From the observed level of agreement between experimental data and simulation results, the slip transfer through the different types of grain boundaries was assessed.

This work was performed under the NSF/DFG Materials World Network initiative under Contracts NSF-DMR-1108211 and ZA523/3-1.

[1] C. Zambaldi, Y. Yang, T. R. Bieler, D. Raabe, J. Mater. Res. **27**, 356 (2012), doi: 10.1557/jmr.2011.334
[2] C. Zambaldi, D. Raabe, Acta Materialia. **58**, 3516 (2010), doi: 10.1016/j.actamat.2010.02.025
[3] C. Zambaldi, C. Zehnder, *in preparation*
[4] C. C. Tasan, M. Diehl, D. Yan, C. Zambaldi, P. Shanthraj, F. Roters, D. Raabe, *submitted to Acta Materialia*

Building realistic atomic models of kerogen using Hybrid Reverse Monte Carlo simulations

Colin Bousige[1,2], Benoît Coasne[1,2], Franz-Josef Ulm[1], Roland Pellenq[1,2]

[1]Department of Civil and Environmental Engineering, Massachusetts Institute of Technology, 77 Massachusetts Avenue, Cambridge MA 02139, USA
[2]<MSE>2, UMI 3466 CNRS-MIT, 77 Massachusetts Avenue, Cambridge MA 02139, USA

Gas and oil shale are unconventional deposits of hydrocarbons that represent important reserves worldwide. Organic-rich shales are natural composites as they consist of different inorganic minerals and a disordered porous carbonaceous material, called kerogen. Understanding and predicting the adsorption, transport, and mechanical properties of such disordered carbons requires having realistic atomistic models that can be used to establish reliable structure – property relationships. Such models can be obtained using Hybrid Reverse Monte Carlo (HRMC) simulations [1,2] in which a numerical atomic model having a structure factor identical to the experimental structure factor is built while using a force field to describe the chemistry of the material.

In this presentation, we will report realistic models of various kerogens obtained using HRMC. We will first show that this HRMC procedure can be computationally accelerated and, hence, applied to much larger systems by combining it with Molecular Dynamics simulations [3]. Thanks to the use of the REBO force field [4], these HRMC can be used to generate numerical models of kerogens with different chemical compositions and maturities. After standard characterization of the different structures in terms of pore size distributions and chemical analysis, the atomistic models of kerogen will be tested against a panel of measurements, accessible to both experiments and molecular simulations, such as nitrogen and water adsorption isotherms, vibrational density of states, and mechanical properties. These refined atomic models will also be used to test the importance of heteroatoms on the hydrophilicity of kerogen.

[1] R. McGreevy and L. Pusztai, *Mol. Sim.* **1**, 359 (1988)
[2] S. Jain et al., *Langmuir* **22**, 9942 (2006)
[3] C. Bousige et al. *To be submitted*
[4] B. Ni et al., *J. Phys. Cond. Mat.* **16**, 7261 (2004)

Integrated Computational and Experimental Structure Determination for Nanoparticles

Min Yu, Andrew B. Yankovich, Amy Kaczmarowski, Dane Morgan, Paul M. Voyles

University of Wisconsin - Madison, Madison, Wisconsin 53706, USA

Advanced experimental techniques, such as scanning transmission electron microscopy (STEM) provide structural images of materials at atomic resolution. However, a single image provides only a two-dimensional projection of the structure, and three-dimensional tomographic imaging at atomic resolution and single-atom sensitivity remains extremely difficult. Experimentally driven structural refinement approaches typically rely on minimizing the error between forward simulation from atomic models and the experiment data. Such optimizations are challenging with limited data and rely on knowing good initial guesses for the ground state, which may be insufficient for predicting metastable, out of equilibrium states. Such optimizations also typically make no direct use of information about the energy of the potential structures. Genetic algorithms (GAs) have proven to be extremely effective in structure prediction for a wide range of complex structures, including clusters, crystals, and grain boundaries, and have also been used to accelerate matching STEM data to a nanoparticle of known structure. We will discuss development of an integrated GA optimization tool that can reverse engineer the 3D structure of a nanoparticle by matching forward modeling to experimental STEM data and simultaneously minimizing the energy. This tool integrates the power of GAs for complex optimization and utilizes both available experimental and energetics data simultaneously. We demonstrate this nanostructure determination tool on STEM data of Au nanoparticles.

Towards efficient fatigue simulation and parameter identification using models with multiple time scales

Guillaume Puel, Denis Aubry

Laboratoire MSSMat (Ecole Centrale Paris/CNRS UMR8579), Grande voie des vignes, 92290 Chatenay-Malabry, France

When dealing with the numerical prediction of material fatigue, classical life estimation methods, such as those described in [1], can give poor results as far as one is interested with complex loading histories (such as a loading with a slow-evolving average) or inertia effects (for high frequency loadings). It seems indeed more relevant for such cases to use time transient models describing how internal variables, such as plastic strain, or isotropic damage, change with respect to time. However, the computational cost associated with such simulations can become prohibitive if each individual cycle has to be computed.

Therefore a specific method is proposed here to drop the calculation cost by factors up to several thousands: it relies on periodic time homogenization, which is similar to what is usually developed in space homogenization. Moreover, the method is based on a sound mathematical framework, which can guarantee the accuracy of the derived equations and solutions. A first formulation of this method has been proposed in [2]: it is based on the assumption that two different time scales (a fast one, associated with the fast cycles, and a slow one) can be defined and decoupled. Using asymptotic expansions of the scale ratio,

it then allows to solve the different equations at the slow time scale only, by taking into account the averaged effect of the fast cycles in the homogenized solution. Since this reference, several extensions have been studied, such as the dynamic framework [3] or how to describe an isotropic damage evolution [4,5].

Moreover, it is possible to address the parameter identification process for such time-homogenized problems. This latter is solved by minimizing a misfit function defined as a norm quantifying the discrepancy between the available measurements and the associated quantities derived from the model [6]. Work is in progress to address the main questions arising when one tries to use in the identification process the time-homogenized model rather than the reference problem.

Eventually, this should lead to a suitable strategy for addressing accurate fatigue predictions and associated parameter identification for arbitrary cases of study, whereas the computational cost remains affordable.

[1] A. Fatemi and L. Yang, Int. J. of Fatigue **20**(1), 9-34 (1998).
[2] T. Guennounni and D. Aubry, Comptes-rendus de l'Academie des Sciences Serie II. **303**(20), 1765-1768 (1986) in French.
[3] G. Puel and D. Aubry, Eur. J. of Comp. Mech. **21**(3-6), 312-324 (2012).
[4] C. Oskay and J. Fish, Int. J. for Num. Meth. in Eng. **61**(3), 329-359 (2004).
[5] A. Devulder, D. Aubry and G. Puel, Comp. Mech. **45**(6), 637-646 (2010).
[6] G. Puel, B. Bourgeteau and G. Puel, Mech. Syst. And Sign. Proc. 36(2), 354-369 (2013).

A coupling method for stochastic polycrystalline models at different scales

Régis Cottereau

Laboratoire MSSMat, École Centrale Paris, CNRS, F-92295 Châtenay-Malabry, France

In this study, we present an approach that allows to couple two stochastic continuum models [1]. In particular it allows to couple a homogeneous (or slowly fluctuating) continuum mechanics model with random constitutive tensor and a polycrystalline model with stochastic anisotropic grains. The latter model represents the micro-scale model while the former would be its upscaled (or homogenized) version. The coupling strategy is performed in the Arlequin framework [2], which is based on a volume coupling and a partition of the energy between two models. The main interest of this framework is that it allows to couple models that are different in nature: discrete vs. continuous, deterministic vs. stochastic, linear vs. nonlinear, with vs. without defects or cracks.

A suitable functional space is chosen for the weak enforcement of the continuity between the two models. The choice of this space ensures that the ensemble average of the two stochastic solutions are equal point-wise in the coupling area, and that appropriate boundary conditions on the stochastic dimension are passed from one model to the other. However, it does not impose any strong continuity, that would induce undue strength localization from one model onto the other. The proposed coupling approach is an extension of a previous work dealing with the coupling of a stochastic model with a deterministic one [3]. It leads to a mixed problem. This choice of functional spaces and coupling operator ensures that the mixed problem has a unique solution that can be approximated by spectral finite elements or a Monte Carlo technique.

This presentation will be focused on showing the interest of such an approach for identification of polycrystalline models when the information comes from experiments performed at different scales. This is for instance the case when measuring concurrently elastic strains through X-ray diffraction during in situ mechanical tests coupled with image correlation. In general, different models are identified at different scales using different experimental information. However, using all the information concurrently and making sure that models at the different scales are compatible (in the sense of homogenization [4]) is a key to relieving some numerical difficulties encountered in the identification process.

Extension to the coupling of dynamics of dislocation models and polycrystalline models will be highlighted if time permits.

[1] Y. Le Guennec, R. Cottereau, D. Clouteau, C. Soize, Prob. Eng. Mech. **Accepted** (2014).
[2] H. Ben Dhia, Comptes Rendus de l'Académie des Sciences de Paris. **326**, 12 (1998).
[3] R. Cottereau, D. Clouteau, H. Ben Dhia, C. Zaccardi, Comp. Meth. Appl. Mech. Eng. **200** (2011).
[4] R. Cottereau, Int. J. Numer. Meth. Eng. 95, 1 (2013)