Advanced Characterization to Parameterize Material Properties for Multiscale Design

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Relevance of Multiscale

Pushing performance and optimization to the limits means that understanding, predicting, and controlling engineering and life-cycle dynamics is increasingly dependent on understanding, predicting, and controlling a hierarchy of physical (chemical and biological) mutually interacting phenomena occurring at a wide range of space and time scales (materials, structures, propulsion systems are classical examples). Traditional engineering models deal mainly or exclusively with the continuum scale. Phenomena at lower scientific scales (meso and micro) are taken into account (when it happens) only through averaging and homogenization procedures.

The basic paradigm is that engineering (macroscopic) space and time scale behaviour of physical and technological systems can be expressed in terms of hierarchies of variables and processes at different space and time scales and inter-scales interaction rules.

Multiscale entails the integrated and synergistic use of different computational, analytical, and experimental techniques with different degree of space and time resolution which, until now, have been developed and applied in different stages of the R&D and engineering process following a fragmented vision and strategy. A fundamental goal is to establish rigorous theoretical connections between macroscale, mesoscale, microscale, and nanoscale models.

Multiscale is the key concept, theory, and method to establish rigorous and comprehensive links between Science and Engineering and put the bases to develop “Unified Visions” and “Unified Planning Strategies” for research, development, and engineering processes.

In order to feed multiscale design with proper parameters describing material properties, characterization and testing metodologies must be deeply revised.

Concerning materials characterization, the following consideration apply: in a Hierarchical Multiscale Materials Characterization approach, "input" and "output" represent, respectively, information received from a lower scale and information transmitted to the higher one.
In Atomistic length-scale modeling, where Characteristic Length is 10^{-9} – 10^{-7} m Characteristic Times 10^{-14}–10^{-10} input are interatomic potentials (calculated with quantum mechanics) and output are dislocation generation, motion and interaction with other defects; scale physics is at the properties of individual defects (dislocations, vacancies, interstitials, dopants), defects mobility, diffusion, clusters, surface reactions.
At microscale length scale modeling (Characteristic Length 10^{-8} – 10^{-6} m, Characteristic Times 10^{-11}–10^{-8} s) input are dislocation generation, motion and interaction with other defects while output are yield and hardening rules for single crystals, with a scale physics related to small ensembles of lattice defects at length scale below the grain size, defect interactions, precipitates, dislocation reactions, the early stages of void growth (which is a dominant failure mechanism in metals and alloys), grain boundaries and the interactions between dislocations and grain boundaries.
Mesoscale Modeling (Characteristic Length 10^{-7} – 10^{-4} m Characteristic Times 10^{-9} – 10^{-3} s) consider as input yield and hardening rules for single crystals to obtain as an output mesoscale models of polycrystal aggregates (100s of grains); in this case scale physics refers to ensembles of lattice defects at length of grain size, shear band, dislocation walls, disclinations, collective dynamics of microstructure, interface diffusion, grain coarsening, recrystallization, crack growth, fracture.
Finaslly, Mesoscale Homogenization / Continuum Model with characteristic Length > 10^{-3} m Characteristic Times >10^{-3} s uses input from mesoscale models of polycrystal aggregates (100s of grains) to generate pressure and strain path dependent yield surface for continuum code (scale physics : polycrystal plasticity, temperature fields, hydrodynamic motion, textures, microstructures homogenization, anisotropic hardening…).
A couple of cases, where parametrization of material properties are carried out by tailoring and enhancing conventional and new characterization tecniques are shown.

Correlating hardness of multilayer films with predictive algoritms.

TiCN coatings on the market today are in general multi-layer TiN/Ti(C_x N_{1-x}) coatings. Such multilayered film configuration enables optimisation of the filmysubstrate as well as the filmyworked material interactions, by controlling the internal stress state, fatigue toughness, hardness and superficial composition of these Ti(C_x N_{1-x}) coatings. This paper presents the results of the investigations on a wear resistant coating made by alternate layers of TiN and Ti(C_x N_{1-x}) (nominal 0.5 mm each), deposited on S600 tool steel by reactive cathodic arc evaporation using a reactive gaseous mixture of methane and nitrogen. Microstructural and compositional characterisation were carried out using ball crater tests, Optical Microscopy, Scanning Electron Microscopy associated with Energy Dispersive Microanalysis and Image Analysis. Micro hardness measurement were evaluated by means of the Chicot and Lesage volume law of mixture model to cope with the problem of the multiple influence of the different layers and the substrate on the real multilayer surface hardness. In order to predict the surface hardness, the model needs the knowledge of the properties of each type of layers and of the substrate (Young modulus, *H*s hardness at infinitely small load and strain hardening coefficient, or ISE index). These properties were measured using microindentation tests from ad hoc samples of single TiN 0.5 mm layer film, single Ti(C_x N_{1-x}) 0.5 mm layer film and the uncoated substrate. Young modulus for the TiN and Ti(C_x N_{1-x}) were evaluated with load–displacement nanoindentation tests; Young modulus for the substrate is from manufacturer. Thickness, composition profiles and microstructure of each film were used to qualify the data input for the model. Experimental measurements on the composite surface hardness allowed then the verification of the predictions.

Developing wear resistant multilayer for severe environment, by correlation of parametric indicators:

Nitride of transition metals (TiN, ZrN, CrN...) are well known for their excellent mechanical and chemical properties and they are actually widely used as wear resistant coating layers in a variety of applications involving hostile environment When particularly severe operating conditions exist, the presence of coating is often useless below a critical thickness, but above the critical thickness the lifetime of components exposed to the harsh environment is considerably increased. However, thickness of PVD coatings is usually limited to about 3-4 microns, due to the increase of inner stresses during film growth and the resulting residual stress, which usually lead the coating to fail in the very early stage of operation. In order to overcame this limitation, taking into consideration previous experiments and theoretical forecast, a set of multilayered Ti/TiN coatings with thickness up to 13 microns were deposited using an arc reactive PVD, with different Ti/TiN thickness ratio and different overall thickness The effectiveness of the thick multilayers Ti/TiN deposited, concerning wear resistance and reliability in the severe operating conditions was experimentally tested. Comparison among different multilayer formulations were also carried out based on:

Overall and single layers thickness Ti vs. TiN ratio Number of layers; in order to find out the best multilayer/deposition process combination, following Quality Indexes have been used: surface defects, roughness, hardness, residual stress measurements, wear test, coating interfaces, surface defects and roughness characterization were made by Digital Optical Microscopy coupled with Image Analysis, SPM, SEM-EDS and FIB.

Residual stresses were investigated by the use of X-ray diffraction microscopy with the $\sin^2\psi$ method. Hardness values were obtained by microindentation (Vickers). Wear behavior were investigated with the rotating wheel test.

In spite of their thickness, Ti/TiN multilayer coated samples revealed residual stress and wear coefficient comparable or even better than the one showed by a single thin (<<13 mm) layer coated samples. Thus the lifetime of thick multilayer

is remarkably higher than single thin film, because comparable high hardness (i.e. wear coefficient) is coupled with comparable high reliability.

The parametrization carried out allowed to find the correlation among process parameters and performance of the coating. The same parametrization allows the multiscale design where relevant issues of different scales could be embedded in simulation codes.