1st International Conference on

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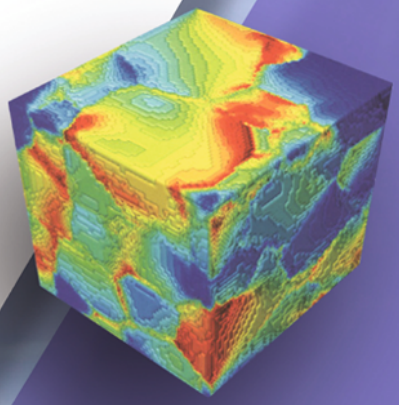
3D Materials Science, 2012



July 8-12, 2012 • Seven Springs Mountain Resort
Seven Springs, Pennsylvania, USA

Conference Proceedings

Edited by:

Marc De Graef
Henning Friis Poulsen
Alexis Lewis
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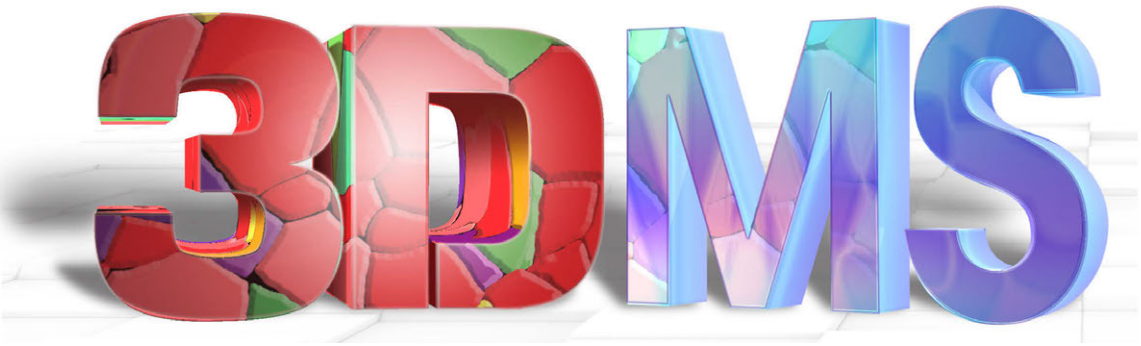

Proceedings of the

1st International Conference on 3D Materials Science

Held

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Seven Springs Mountain Resort

Seven Springs, Pennsylvania, USA

Edited by

Marc De Graef, Henning Friis Poulsen, Alexis Lewis,

Jeff Simmons, George Spanos

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ISBN 978-3-319-48573-7 ISBN 978-3-319-48762-5 (eBook)
DOI 10.1007/978-3-319-48762-5

Chemistry and Materials Science: Professional

Published by Springer International Publishers, Switzerland, 2016
Reprint of the original edition published by John Wiley & Sons, Inc., 2012, 978-1-11847-039-8

Printed on acid-free paper

This Springer imprint is published by Springer Nature
The registered company is Springer International Publishing AG
The registered company address is: Gewerbestrasse 11, 6330 Cham, Switzerland

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Preface

This volume represents a collection of papers presented at the first International Conference on 3D Materials Science, a specialty conference organized by the Minerals, Metals & Materials Society (TMS) and the conference organizers, and held at Seven Springs Mountain Resort, Seven Springs, Pennsylvania, USA from July 8-12, 2012.

The aim of the first International Conference on 3D Materials Science was to bring together world leaders in the field, at a time when 3D materials characterization and analysis techniques are moving beyond initial qualitative measurements and into an era where full 3D quantification of microstructures is becoming the norm. While many of the early challenges of data collection have been overcome, either by the development of new software and hardware or by automation, new techniques are steadily being introduced and improved upon, and many challenges in the processing, storage, sharing, and analysis of 3D data exist. This workshop sought to bring together the community to share ideas and tools, and to roadmap the future directions of 3D Materials Science.

The conference covered research in Applications of 3D Experimental Techniques across Length Scales (including both Destructive and Non-Destructive techniques), Image Processing of 2D and 3D Microstructural Data, Digital Representations of 3D Microstructures, Storage and Sharing of 3D Data, Microstructure-Property Relationships in 3D, 3D Interfaces and Microstructural Evolution, and Future Directions in 3D Materials Science. The conference included a plenary session and 20 invited presentations from prominent international speakers working in the research areas listed, nearly 60 oral presentations, and nearly 100 poster presentations including a special student poster session. The International Advisory Committee represented 11 countries, and the speakers and poster presenters represented 25 different countries, making this a truly international conference.

This proceedings collection contains high-impact research contributions representing the poster, oral, invited, and plenary sessions. The high quality of the research presented at the conference is reflected in these manuscripts. It is the aim of editors of this volume, the conference organizers, and TMS, that this conference, and the proceedings contained herein, serve to bring together the 3D Materials Science community to maximize cooperative interaction and advance the field in a meaningful way.

About the Conference Editors/Organizers

Marc De Graef

Professor Marc De Graef is Professor of Materials Science and Engineering at Carnegie Mellon University in Pittsburgh, PA. He joined the faculty in March 1993 after a post-doctoral period in the Materials Department at the University of California at Santa Barbara. He received his BS and MS degrees in physics from the University of Antwerp (Belgium) in 1983, and his Ph.D. in physics from the Catholic University of Leuven (Belgium) in 1989. He is co-director of the J. Earle and Mary Roberts Materials Characterization Laboratory. He served on the Board of Directors of TMS, received the 2012 TMS Educator Award, and is a Fellow of the Microscopy Society of America. His research interests lie in the area of microstructural characterization of structural intermetallics and magnetic materials, with a strong emphasis on 3-D characterization. His experimental approaches include scanning and transmission electron microscopy, focused ion beam microscopy, and x-ray and electron tomography. On the theoretical side he has an interest in the physics of shapes, and in the quantitative simulation of electron scattering imaging modalities.

Henning Friis Poulsen

Professor Henning Friis Poulsen joined the section for “neutron and x-ray materials physics” at the Technical University of Denmark in January 2012. Prior to this, Poulsen was a research professor at the Risø National Laboratory for Sustainable Energy in Denmark, where he headed the Centre of Excellence: “metalstructures in four dimensions”. His research has focused on the development of synchrotron and TEM based 3D and 4D characterization techniques and their applications within materials science and engineering, in particular metallurgy. He currently holds an Advanced Grant from the European Research Council. Poulsen was granted a Dr. Techn. degree from the Technical University of Denmark in 2004. He received his PhD in Physics from the University of Copenhagen, his MS in Engineering from the Technical University of Denmark and his BS in Mathematics and Physics from the University of Copenhagen.

Alexis C Lewis

Dr. Alexis C. Lewis is a staff scientist at the US Naval Research Laboratory in Washington, DC, where she began as a National Research Council Postdoctoral Associate in 2003, and was promoted to Materials Research Engineer in 2005. Her research focuses on three-dimensional characterization of materials, with a focus on 3D crystallography, and measuring structure-property relationships through experiment and simulation. Dr. Lewis is the former chair of the TMS Advanced Characterization, Testing, and Simulation committee, as well as a member of the Mechanical Behavior and the Women in Materials Science and Engineering Committee of TMS. She is a member of TMS, ASM and MRS. Dr. Lewis received her SB in Materials Science and Engineering from MIT in 1997, and her PhD in Materials Science and Engineering from the Johns Hopkins University in 2003.

Jeff Simmons

Dr. Jeff Simmons is a scientist at the Materials and Manufacturing Directorate of the Air Force Research Laboratory. He has a B.S. degree in Metallurgical Engineering from the New Mexico Institute of Mining and Technology and M.E. and Ph.D. Degrees in Metallurgical Engineering and Materials Science and Materials Science, respectively, from Carnegie Mellon University. After receiving his Ph.D. in 1992, he joined the Materials Directorate at Wright Patterson AFB as a post doctoral researcher and as a research contractor. In 1998, he joined the Air Force Research Laboratory as a research scientist. During his tenure, he has worked on both basic research as well as engineering applications in the aerospace industry. His research interests are in 3-D Materials Science, particularly in development of advanced algorithms for production and analysis of large image datasets. He has published and has organized symposia in the Materials Science and Signal Processing fields on applications of electronic imaging algorithms towards the analysis of digital microscopy. Other research interests have included mathematical representations of microstructures, mesoscale modeling of microstructure development (Phase Field), atomistic modeling of defect properties, and computational thermodynamics. Non-research experience has included leading teams to develop analytical tools for digital data, integration of computer resources for materials simulations, computer security, and network design as well as manufacturing, particularly in machining applications. He has overseen execution of Air Force contracts in Integrated Computational Materials Science and Engineering (ICMSE). Dr. Simmons is a member of the TMS committees of Advanced Characterization, Testing, and Simulations (ACTS) and has served as its chair, Phase Transformations, and Integrated Computational Materials Engineering (ICME). He is a member of TMS, ACM, and the IEEE Computer and Signal Processing Societies.

George Spanos

Dr. George Spanos, TMS Technical Director, is responsible for the technical direction of TMS, located at the society's headquarters in Warrendale, PA. In addition, he contributes to the development and execution of the society's strategic plan. He received his B.S. (1982), M.E. (1985) and Ph.D. (1989) degrees in Metallurgical Engineering and Materials Science from Carnegie Mellon University. In 1989 he joined the Naval Research Laboratory (NRL) as a staff scientist, in 1994 was promoted to Section Head (Microstructural Evolution Section) at NRL, and in June of 2010 joined TMS as their Technical Director. Dr. Spanos is author/co-author of 92 technical publications which have been cited more than 1,500 times, in the field of phase transformations, processing-structure-property relationships, 3D materials analyses, and ICME. Some of his past and present professional affiliations include: member of the Board of Governors of Acta Materialia Inc. (2008-2010), past chairman (1999) and member (1996-1999) of the Joint Commission for Metall. and Materials Trans., Chairman (1995-1996) and Key Reader (1992 - present) of the Board of Review of Metall. and Materials Trans. A, and member of a number of TMS committees. Some of his awards include: Fellow of ASM-International (2004), Marcus A. Grossman Award for best article in Metall. and Mat. Trans. for authors under 40 (2001), two Technology Transfer Awards at NRL (2000, 2005), NRL 2009 Commanding Officer's Award for Achievement in Equal Employment Opportunity (EEO).

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TMS (The Minerals, Metals & Materials Society), 2012

PERSPECTIVES ON MATERIALS SCIENCE IN 3D

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Keywords

3D techniques, 3DXRD, recrystallization, challenges for 3D science, success of 3D science

Abstract

Materials characterization in 3D has opened a new era in materials science, which is discussed in this paper. The original motivations and visions behind the development of one of the new 3D techniques, namely the three dimensional x-ray diffraction (3DXRD) method, are presented and the route to its implementation is described. The present status of materials science in 3D is illustrated by examples related to recrystallization. Finally, challenges and suggestions for the future success for 3D Materials Science relating to hardware evolution, data analysis, data exchange and modeling are discussed.

Materials Science in 3 and 4D

The Materials Science theme generally aims at linking materials processing with materials properties and performances. The key to understanding this link is typically in the materials microstructure, and microscopy, in particular electron microscopy, is by far the most valuable experimental tool for microstructure investigations. All the microscope techniques are by nature 2D (x, y) techniques which actually limit the microstructure information obtained by these methods and have led to misinterpretations and mistakes [e.g. 1]. Transmission electron microscopy does allow 2 ½ D (x, y, thin z section) characterizations both by tomography and diffraction techniques [e.g. 2-4].

Another third dimension, namely time, t, has been studied by electron microscope techniques (x,y,t), so the evolution of a specific microstructure during for example deformation or annealing has been studied by implementing stress rigs and furnaces in the microscope [e.g. 5-7]. Such measurements clearly reveal the microstructural evolution in the sample surface, but uncertainties of course always exist if the observations are affected by the free surface, and bulk phenomena occurring below the inspected surface may suddenly appear – e.g. a nucleus may

form below the surface and grow to be seen in the inspected surface. So clearly 3D (x, y, z) and 4D (x, y, z, t) techniques are the way forward for many types of experiments.

3D techniques are not new. Serial sectioning has been used for decades [e.g. 9, 10], x-ray methods have long been used for local bulk characterization [e.g. 11, 12] and neutron diffraction has been used to determine for example average textures and residual stresses in large bulk sample volumes [e.g. 13, 14].

What is new is that within the last 10-20 years, the scientific community has really realized the need for 3D and 4D techniques and has devoted significant efforts to advance the existing 3D/4D methods and to develop new unique techniques. The goals in these efforts include: much easier, less manpower-requiring operations; better spatial resolution; non-destructive methods; fast measurements. This has led to a whole suite of modern 3D and 4D techniques, and 3D/4D results are reported in more and more publications. The techniques behind the most frequently published 3D/4D results are in two groups: advanced serial sectioning and x-ray methods. The serial sectioning methods encompass semi-automatic and fully automatic mechanical sectioning, which microstructural inspection in optical as well as scanning electron microscopes [e.g. 15-18], high resolution focused ion beam sectioning in the scanning microscope [e.g. 19-20] and sectioning by lasers [21]. The new x-ray methods include three dimensional x-ray diffraction (3DXRD) for fast non-destructive orientation measurements [e.g. 22-24] and 3D polychromatic x-ray micro diffraction for high spatial resolution orientation measurements [25-27] as well as techniques where slits[28] and collimators [29] are used to get the third dimension.

3DXRD – From Idea to Implementation

The vision was to develop a technique allowing non-destructive measurements in selected local *μ-sized* volumes within the *bulk* of mm^3-cm^3 samples. It was motivated by the need to study local deformation and recrystallization phenomena occurring in the bulk *in-situ,* while samples were deformed or annealed. For example for recrystallization (which may be considered as a very well known and well documented process, which is used intensely in the industry) it is remarkable that real key phenomena are not known, e.g. where do the nuclei form and with what crystallographic orientations, how do the annealing-induced boundaries surrounding the nuclei and recrystallizing grains migrate and interact with all the deformation-induced dislocation boundaries and what parameters are of importance here.

A basic requirement for developing a technique to fulfill the described vision is a beam which can penetrate deeply (>1 mm) in materials and which is so intense that even diffraction signals from μ-sized volume elements can be distinguished from the background. High energy x-rays from synchrotrons provide such a beam. When the energy of the x-rays is about 80 keV, the penetration depth (defined as the depth at which the transmitted intensity is 10 %) is 5mm in steel and 4 cm in aluminium. A very intense beam on the sample is obtained by focusing.

For us the route towards the implementation of the vision started by hiring an expert in high energy x-rays (Henning F. Poulsen), who collected the scientific ideas and together with the scientists behind the ideas started to define specifications etc. for the dream 3DXRD instrument. The first paper describing the idea behind the 3DXRD microscope and the first set of preliminary data was published already in 1995 [22]. During the following years, various beam lines at HASYLAB in Hamburg, Germany and at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France were used for further development of the 3DXRD technique and for testing the equipment for different types of experiments [e.g. 30]. Following a workshop held in February 1998 at ESRF on the use of the 3DXRD technique it was decided to build a dedicated hutch (measuring area) for a permanent installation of a 3DXRD microscope at the Materials Science beamline ID11. The agreement was that ESRF should build the hutch with all the necessary installations, while Risø should develop, build and install the microscope. The optics should be developed as a joint-venture. After completion, the 3DXRD microscope should become an ESRF instrument in the sense that potential users may apply for beamtime on it using the regular ESRF application procedure, and that the instrument, therefore, is available to anybody interested having a qualified research proposal.

The 3DXRD microscope was commissioned during the summer 1999 and has been in regular operation ever since. During the recent years, it has been upgraded to obtain finer spatial resolution including moving it to a new experimental hutch. Our group now is not directly involved in the operation of the 3DXRD instrument. We contribute with ideas, some software development and as “large” users via a series of approved so-called long-term proposals, which have allowed us around 4-6 weeks beamtime per year since year 2000.

The described process (from instrument builders to large users) is believed to be optimal as the best operation of beamline instruments is by scientists at the synchrotron and our role is of course to be materials scientists using the 3DXRD for key experiments and ambassadors helping the ESRF staff spreading the knowledge about the 3DXRD’s experimental possibilities.

Need for 3D and 4D Tools in Recrystallization Studies

Nucleation of recrystallization is very difficult to study experimentally; the nuclei are small and few, so large data sets are typically needed to get statistically sound data. Furthermore, if the issue is to study nucleation sites or orientation relationships between nuclei and parent site in the deformed sample, static 2D measurements suffer from the so-called “lost-evidence” problem [31], namely that when a nucleus is observed with a 2D (x, y) technique, it is impossible to see what was there before the nucleus formed so the exact nucleation site or orientation relationship is unknown. Also results obtained by in-situ annealing measurements in e.g. a SEM (x, y, t) may be problematic to interpret due to surface effects.

So far, few studies have been published on 3D/4D observations of nucleation: The relationship between nuclei and large second phase particles have been quantified by careful serial sectioning of Al-1.2 wt Mn [32] and Al 3004 [18]. In both cases results obtained by 2D methods were confirmed namely that large particles are preferential nucleation sites. However, the agreement is only qualitative in the sense that 2D observations lead to quite different quantitative results than 3D [18]. Serial sectioning was also used to map the spatial distribution of nucleation sites [17], and the results were used as input for modeling of the effects of nuclei clustering on the recrystallization kinetics and grain size distribution. It was shown that even weak clustering has clear effects on both recrystallization kinetics and size distribution [33].

In order to predict the recrystallization texture and understand the various nucleation mechanisms, it is important to know which crystallographic orientations the nuclei have at the various nucleation sites. It is generally assumed that nuclei form with orientations present at the nucleation sites in the deformed state. However, other publications report on formation of nuclei with new orientations which are not seen in the deformed microstructure [e.g. 8, 34-36]. With the classical 2D or the serial sectioning 3D method one can only guess which orientations were present at the exact nucleation sites before the annealing.

Only 4D investigations can quantify if nuclei may form with orientations different from those present at the nucleation sites prior to annealing. 3DXRD has been used for such investigations [37, 38] and it was shown that nuclei with new orientations do form and their orientations cannot be explained by twinning up to second order. The data were analyzed for relationships between the new nuclei orientations and the rotation axis across deformation induced dislocation boundaries in the deformed parent grains. The results suggest that the origin for the formation of nuclei with new orientations relates to the misorientation distribution in the deformed parent grain and that the misorientation axes between a nucleus and the parent grain may be identical to a misorientation axes across dislocation boundaries within that grain [38].

During recrystallization the grains grow by boundary migration through the deformed matrix. Whenever two grains impinge, the boundary motion will stop. Further migration would be defined as grain growth. Generally, it is supposed that the velocity of boundary motion, v, is proportional to the driving force F as expressed by the relationship:

$$v = M F$$

where M is the mobility. For recrystallization, the driving force F is assumed to be given by the stored energy in the deformed microstructure (F_D) which typically is orders of magnitude larger than the driving force related to boundary curvature during grain growth. Whereas several grain growth experiments have confirmed the validity of eq (1) [e.g. 39, 40] much less is done for recrystallization. The few recrystallization experiments focusing on this issue have all been done by static stereological 2D characterizations which either confirm eq (1) [e.g. 41, 42] or found a power-law relationship between v and F [43], i.e. another simple relationship.

In-situ 2D investigations during recrystallization as well as ex-situ 2D characterizations of the same surface area after a series of consecutive annealing treatments, however reveal very complex boundary migration and velocities which locally deviate significantly from that predicted using average F and M values in eq (1) [7, 44]. Whether this is a real effect or a consequence of the 2D character of the experiment cannot be proven. 4D (x, y, z, t) experiments are required.

The migration of a boundary surrounding a single recrystallizing grain through a weakly deformed single crystal matrix has been studied in-situ by 3DXRD [45]. These measurements also reveal a very complex boundary migration process. It is found that:

1) Flat facets may form, which migrate forward at the same rate for extended periods of time.

2) The non-faceted segments of the boundary do not migrate at a constant rate through the relatively homogeneous deformed matrix of the single crystal. On the contrary, segments of the boundary move forward for a while then stop, move again etc. (stop-go motion).

3) Local protrusions and retrusions typically form locally on the migrating boundary.

As these results are observed directly in 4D, they cannot be due to experimental artifacts. Standard recrystallization theories and models (e.g. eq (1)) do not describe the 4D results well and have to be revised. For this purpose the 3DXRD measurements have to be supplemented by detailed microscopical investigations in order to understand what is going on. An example of some of this work is given elsewhere in these proceedings [43]. Also an experiment has been performed using the 3D polychromatic x-ray micro diffraction method at APS. This technique has a much better spatial resolution than the 3DXRD and thus allows for direct observations of effects of the local deformed microstructure on migration of recrystallization boundaries, which is considered instrumental for establishment of improved recrystallization theories and models.

Challenges and Suggestions for the Future Success of 3D Materials Science

Indication of success for 3D/4D characterizations already exist; i) results have been published which prove that 2D is not enough and some new physical mechanisms have been suggested based on 3D/4D data, ii) several of the experimental techniques have been further advanced and can now be considered as relatively mature and iii) the 3D/4D community is growing from the initial stage when it was mostly the group(s) behind a given technique, who was using that technique.
There is still, however, a long way to go before 3D/4D will be widely used. This is not unusual, and history has shown that some degree of commercialization often is needed. The electron back scattering pattern (EBSP) technique for example was developed over many years in individual

universities/national laboratories but not until two companies were created focusing on the EBSP method and in particular the data analysis, EBSP became a "need to have" technique in most materials science laboratories in academia and industry. Clearly of course the companies realized correctly that the EBSP technique fulfills a general scientific need of sufficient importance to base a business model on. And the companies they contributed significantly to the field by developing easy, user-friendly and fast experimental measurements and even more important, they developed systems for easy data analysis and beautiful data presentation.

Little commercialization has yet been realized for the 3D/4D methods, except from the RoboMet which is based on mechanical serial sectioning combined with optical microscopy and FIB in the SEM. In my opinion, this is not because there is not sufficient scientific need for the 3D/4D methods – some users may have been scared off because of the present complexities with the methods, but the need *is* there.

In the following some major obstacles and suggestions for the future shall be discussed.

1. There are many principally different 3D/4D methods

One data analysis system can therefore not be developed which serves them all. The raw data analysis and e.g. the alignment procedure needed to get serial sections in register are different. So this part of the data analysis clearly has to be developed by the instrument builders or in very close collaboration with these. From thereon, however, the 3D/4D data analysis and representation could (should) be united. The data sets are large, which may be a challenge in its own right, but other communities – in particular the game industries do handle far larger sets, and it might be an idea to learn from them. The whole 3D/4D materials society would benefit, if a company would take up this challenge (or the society could unite to work on just one representation system). Another challenge here is that in present days' publication channels, it is hard to visualize 3D/4D data. This would mean that in the papers we do not only have to focus on pretty pictures but also on specific scientific points; which may actually be an advantage for this field.

2. The 3D/4D community is scattered

The scientific focus is broad, which this conference also illustrates. It might be an advantage to keep the community close together, not only by meeting at conferences, but also via establishments of networks. This should be networks focusing on technique development, on science-technique questions/possibilities and on data representation and visualization. Web-based sharing of information and tools may be a way forward.

3. Some of the 3D/4D methods only operate at large international facilities

Here it is either very costly to get beamtime or if the usual peer-reviewed beamtime application procedure is used, it is hard to get beamtime – success rates below 10% is not unusual and in all

cases the time from idea to experiment is very long. This clearly limits the widespread use of these 3D/4D methods, and it is expected that this will always be the case in spite of the fact that more large scale facilities are being built. One might think of making some of the measurements faster more standard, so some kind of block allocation system might be put into place as parts of the biology community have done. But generally, an advice is only to use the 3D/4D techniques at the large scale facilities when it is really needed, i.e. the results cannot be obtained in any other way and always be very well prepared with optimal and well characterized samples for the 3D/4D experiment at the large scale facility.

4. Data analysis takes months, if not years

This is in particular true for the 3D/4D technique at the large scale facilities. Some *on-line* data visualization is extremely important, which can guide the next measurements and the user can avoid losing some of the precious beamtime on useless data. This has been implemented on some of the beamlines, e.g. on APPS 34-ID for the 3D polychromatic x-ray micro diffraction technique. Easier data analysis may be a compromise between constantly improving the experimental techniques to reach new possibilities and keeping to some fixed standards, which can be used for series of measurements. Also here establishment of communities between technique developers and scientific users could help getting this balance right.

Concluding Remarks

The 3D/4D journey has so far been fantastic. Occasionally, it has been hair-raising to look inside the materials and see things which have hitherto been hidden and not possible to observe with other techniques. We are, however, in my opinion now at a critical stage, where we have to bring 3D/4D investigations beyond the demonstration stage and produce series of scientifically important results, which in term will stimulate the modeling and physical understanding. Challenges include firm establishment of the 3D/4D community(-ies), treatment of huge data sets, capturing methods to visualize the data and extract the scientifically important information, prescribing joint protocols to exchange data, not only between the different techniques, but also between experiments and models, i.e. creating a lively database, which stores all the data, keeps track of their associations and evolves as new experiments or modeling results are available. With the goal of overcoming these challenges it seems necessary to join not only the 3D/4D communities but also to create strong links to the ICME (Integrated Computational Materials Engineering) community, which deals with some of the same scientific and industrial needs for materials results which go beyond what can be obtained by 2D experimental methods.

Acknowledgements

The Danish National Research Foundation is acknowledged for funding the Danish Chinese Center for Nanometals, within which this work has been performed.

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1st International Conference on 3D Materials Science
Edited by: Marc De Graef, Henning Friis Poulsen, Alexis Lewis, Jeff Simmons, George Spanos
TMS (The Minerals, Metals & Materials Society), 2012

3D Investigation of Cracking Behavior in a Ni Superalloy

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Abstract

The high temperature fatigue performance of Ni-base superalloys is critical to gas turbine applications and as such, requires a more fundamental understanding when designing and producing turbine components. To investigate the relationship to local microstructure, a fatigue specimen was cycled under conditions designed specifically to result in intergranular propagation. Prior to failure, the test was interrupted and a 3D data set was reconstructed destructively from optical and EBSD slices taken from around the tip of the growing crack. The data set was investigated to understand the character of grain boundary planes along the crack front with respect those of the bulk material.

Introduction

Gas turbines demand exceptional high temperature material performance. Standard operation imposes complex cyclic stress states and corrosive environments, making alloy design crucial to component life. Consequently, it is no misnomer that the class of alloys suited for gas turbine applications are called 'superalloys.' In service Ni superalloys must survive a number of engine cycles in the range of 15,000 or 150,000, depending on the application, and this expected life is currently set through curves generated by thorough mechanical testing. To supplement this data and understand the microstructural effects on life, an effort to explore the three-dimensional nature of a crack tip was initiated. Methodology and preliminary results are discussed here.

Mechanical Testing

A Ni-base superalloy fatigue specimen with rectangular cross section (0.400” wide by 0.168” thick), known as a KB bar, was prepared with a 7-mil deep by 14-mil wide EDM surface flaw in the center of the gage section. An image of a KB bar is shown in Figure 1. The specimen blank was fully solutioned and aged, prior to finish-machining. A room temperature pre-crack sequence was imposed at 10Hz under a constant ΔK of 14.25ksi*in$^{1/2}$ (R = 0.05) to extend a fatigue crack from the EDM starter flaw. The test temperature was then elevated to 1300°F and a trapezoidal cyclic waveform was imposed with 1-second ramps and a 360-second hold at peak stress. This combination of temperature, stress, and waveform resulted in a fully intergranular fatigue failure mode. Crack length measurements were made throughout with a reversing DC electrical potential drop system. The test was interrupted prior to failure, and the specimen was prepared for serial sectioning to analyze the microstructure adjacent to the main crack.

To prepare the sample, a section of the KB bar containing the fatigue crack was mounted in a conductive phenolic compound, ground, and polished in longitudinal cross-section. The grinding was controlled so that the plane of the final polish was approximately transversely centered in the bar, capturing the deepest part of the crack. Additionally, the cross-section was mounted

away from the mount center and a flat was polished into the side of the mount for positioning in the SEM and the optical bench.

Data Acquisition

Once the mount was prepared, a serial sectioning routine similar to [1] was applied to generate data for 3D reconstruction and analysis. First, four Knoop micro-indents were set in a rectangular array surrounding the crack tip using a Clemex JS-2000 micro-indenter. The initial width of each indent was measured and recorded to provide a baseline for measuring material removal. Next the mount was positioned in a Struer's Prepamatic-2 autopolisher and nominally 1.0μm of material was removed. A modified Kalling's etch was applied to delineated the grain boundaries and a multi-image optical montage of the entire crack tip was taken on a Zeiss Axio Observer Z1.m microscope, with a 0.11μm pixel size. The indent widths were measured again after the polish and imaging, and the removal depth was calculated using the geometry of the indent. This sequence was repeated five times, with fresh indents being applied every other slice in a rotating pattern. Once the fifth cycle was completed the flat of the sample was placed in a Camscan 44 W-filament SEM on a 20° pre-tilted wedge with affixed stoppers for accurate sample positioning. This positioning provided a 70° tilt of the sample plane for EBSD mapping. Once the SEM was evacuated, the accelerating voltage was set to 20kV and sample was positioned to map the region of interest. Before scanning, the filament was allowed to equilibrate for a minimum of 3 hours in order to minimize drift during mapping. Finally a 1631x1259 pixel map, with a step size of 0.75 μm, was performed. The map took approximately 13 hours to complete at a scan rate of 44 points/second. The entire sub-process of 5 optical analyses and one EBSD analysis was repeated over the course of 4 months to produce ~150 individual optical slices and ~30 EBSD maps for the 3D reconstruction, schematically shown in Figure 1.

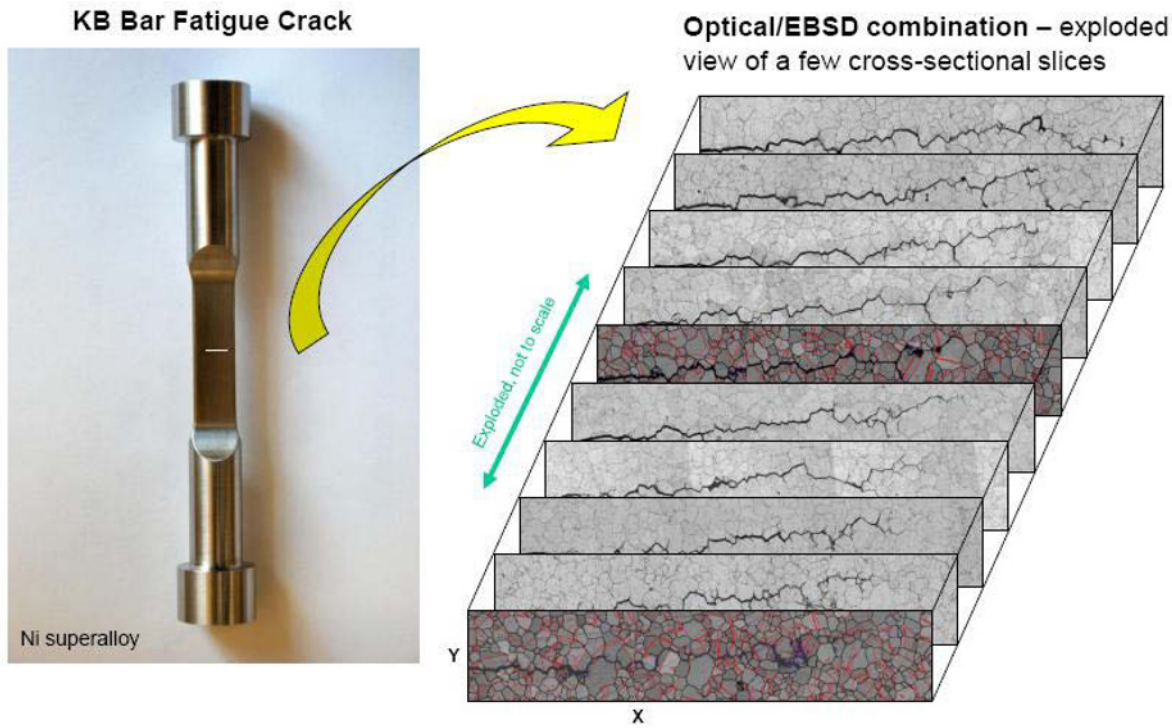

Figure 1 – Schematic representation of KB bar crack 3D reconstruction

Results

Figure 2 shows the removal rate histogram for the 150 slices. The standard deviation of the mechanical material removal was 0.21µm, and the overall average was 1.01µm, within 1% of the target. A typical optical montage and a corresponding EBSD map are shown in Figure 3. The optical montages generally did not reveal twin boundaries. Some are visible, but most are not. However, twins were well captured by the EBSD maps, which will eventually allow for their incorporation into the reconstruction. The percentage of correctly indexing points in the EBSD maps was generally 95% or more. Cleanup was applied to the data to fill in grain boundaries, but care was taken to avoid filling the crack to aid in crack extraction. In Figure 3, ~3.5% of points were generated from the cleanup routine, with red indicating a Σ3 boundary, black indicating a regular grain boundary, green representing the remaining unindexed area.

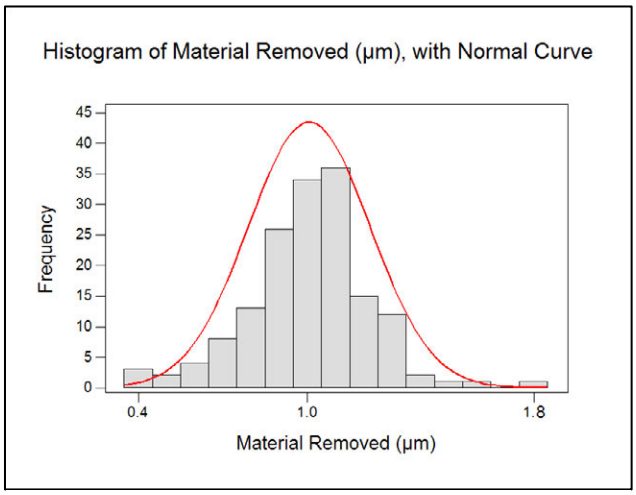

Figure 2 – Histogram of sectioning measurements

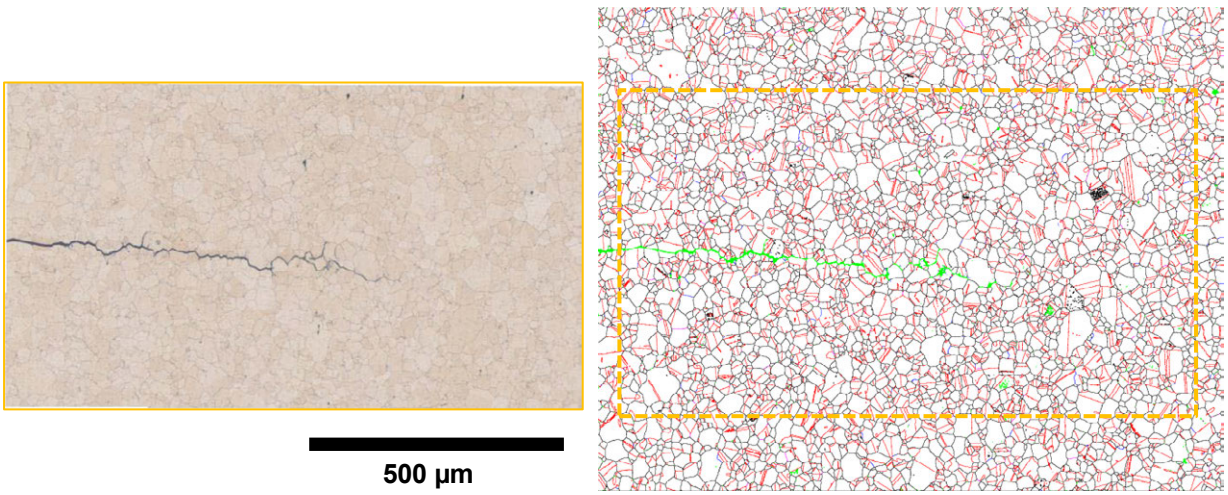

Figure 3 – Typical optical montage (L) and EBSD map (R). Black, red, and green in the map signify regular boundaries, special boundaries, and unindexed points, respectively.

Data Reconstruction

To reconstruct the 3D volume, first the individual optical tiles from each slice were montaged using the Zeiss software. The optical images were then stacked and aligned within the computer using in-house registration routines. The misalignments between the sections primarily consisted of translations, with very small rotational misalignments. The translations were determined by the maximum in the cross-correlation of the images.

Next the EBSD scans were aligned with the optical images. While the task of matching two images of the same identical area might seem easier than aligning different sections, the alignment of the two image modalities proved to be more challenging. One difficulty was that the EBSD images had small inherent distortions. These distortions were likely due to small amounts of drift, minor errors in dynamic focus or electron beam raster alignment to the sample, and some small sample positional errors. For more information on distortions in EBSD maps, see [2]. While careful data collection methods minimized these distortions, when directly comparing small features over a large area, such as the interface of a crack, even small image distortions led to significant misalignment of the images.

To align the two datasets, the optical image was treated as the baseline image, and its sister EBSD image was undistorted to achieve the highest degree of overlap with it. The EBSD image was undistorted using a second-degree polynomial with separate coefficients for the x and y directions, leading to a 12 parameter optimization. The coefficients of the polynomials were determined using a Nelder and Mead optimization algorithm, which maximizes a given generic function given a set of adjustable parameters. The optimized function in this case measured the degree of overlap in the two images using mutual information.

Mutual information is a concept borrowed from the field of information technology which determines probability that a set of pixels contain the same information, regardless of the independent values of the pixels. The mutual information of two images is related to the image entropy, H of the image defined by:

$$H = -\sum_{i=0}^{255} p_i \ln(p_i)$$

where p_i is the probability that a pixel within the image has an intensity of i, within the a range of 0 to 255. The joint entropy of two images, im1 and im2, measures the degree of correlation between the two images and is similar to the concept of the entropy of mixing in solutions, is given by:

$$H(im1,im2) = -\sum_{j=0}^{255}\sum_{i=0}^{255} p_{i,j} \ln(p_{i,j})$$

where pi,j is the probability that if im1 has an intensity of i then im2 has the probability of having intensity j. The mutual information, MI, between the two images is then given by:

$$MI(im1,im2) = H(im1) + H(im2) - H(im1,im2)$$

Thus, if the amount of shared information between the two images increases, the value of the MI will increase, regardless of the exact values of the features within the images. For further information on the use of mutual information for data alignment see [3].

The mutual information between the optical image and the “Band Contrast” image from the EBSD data was maximized, since both of these imaging modes provided strong contrast at the