

# Structure-property relations of wood fibres; 3D characterisation and modelling (Woodfibre3D)

## FINAL REPORT

<b>Title of the research project</b>	<b>Structure-property relations of wood fibres: 3D characteriation and modelling</b>
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<b>Coordinator of the project</b>	Bjørn S. Tanem
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## BASIC PROJECT DATA

<b>Project period</b>	01.01.2008-31.12.2010
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<b>Contact information of the coordinator</b> (institute/unit, address, telephone, fax, e-mail)	SINTEF Materials and Chemistry Høgskoleringen 5 7465 Trondheim, Norway +47 98 28 39 13 Bjorn.s.tanem@sintef.no
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<b>URL of the project</b>	http://[web address]
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## FUNDING

<b>Total budget in EUR</b>	[amount in EUR]
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<b>Public funding from WoodWisdom-Net Research Programme:</b>	Total funding granted in EUR by source:
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<u>Finland</u> Academy of Finland (AKA)	163 400 EUR
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<u>Denmark</u> Danish Research Council for Production and Technology Sciences (FTP)	331 618 EUR3
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Norway

The Research Council of Norway (RCN) 612 514 EUR

Sweden

The Swedish Research Council for Environment,  
Agricultural Sciences and Spatial Planning (Formas) 402 856 EUR

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**Other public funding:**

[Name of the funding organization, Country] [amount in EUR]

[Name of the funding organization, Country] [amount in EUR]

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**Other funding:**

[Name of the funding organization, Country] [amount in EUR]

[Name of the funding organization, Country] [amount in EUR]

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**PROJECT TEAM (main participants)**

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Kristofer Gamstedt,	M	KTH, Dept Fibre and Polymer Technology	FORMAS
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Markku Kataja	M	University of Jyväskylä Department of Physics	AKA
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Gary Chinga Carrasco	M	Paper and Fibre Research Institute	RCN
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Gunilla Borgefors	F	Swedish University of Agricultural Sciences	FORMAS
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Anders Brun	M	Swedish University of Agricultural Sciences	FORMAS
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Cris Luengo	M	Swedish University of Agricultural Sciences	FORMAS
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Bent Sørensen	M	Risø National Laboratory for Sustainable Energy, Technical University of Denmark	FTP
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Bo Madsen	M	Risø National Laboratory for Sustainable Energy, Technical University of Denmark	FTP
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Bjørn S. Tanem                      M              SINTEF Materials and Chemistry    RCN

### DEGREES

Degrees earned or to be earned within this project.

2009	M.Sc.	M	Arttu Miettinen, 1985	Univ. of Jyväskylä	Markku Kataja
	PhD*	M	Arttu Miettinen, 1985	Univ. of Jyväskylä	Markku Kataja
2010	Post.Doc	F	Asuka Yamakawa, 19xx	NTNU	Gary Chinga Carrasco/Øyvind W. Gregersen
2009	MSc	M	Andreas Köhler, 19xx	KTH	Kristofer Gamstedt
	PhD*	M	Mustafa Aslan, 1982	DTU	Bent Sørensen
	PhD*	M	Erik Wernersson, 1984	SLU	Gunilla Borgefors
2008	Licentiate degree	M	Filip Malmberg, 1980	SLU	Gunilla Borgefors
2009	MSc	M	Fabrice Mangiapane, 1986	KTH	Kristofer Gamstedt
2010	MsC	M	Thomas Joffre, 1987	KTH	Kristofer Gamstedt

\*PhD study will continue beyond the ending of Woodfibre3D

## ABSTRACT

*A summary of the project.*

Woodfibre3D is a three-year transnational research project with active participations from Sweden (KTH, SLU), Finland (University of Jyväskylä), Denmark (Risø-DTU) and Norway (NTNU, PFI and SINTEF). One of the main objectives of the project has been to establish feasible procedures and routines to obtain and interpret x-ray synchrotron data of wood fibres and wood fibre reinforced composites with sufficient resolution. When combined with the consortium partner's own lab-scale facilities (table-top x-ray facility, electron microscopy and mechanical testing) and simultaneous development of advanced image processing competence and procedures, this advanced approach is demonstrated to be a powerful tool for establishing structure-property relations of wood fibres and wood fibre reinforced composites. This approach opens for modelling work, having inherent predictive power and importance for material development.

## 1.1 Introduction

### 1.1.1 Background

*Describe the background of the project and the basic problem that it sought to address.*

Traditional manufacturing of pulp for paper products are in a process of moving to countries with plantations of fast growing trees located close to the mills. Hence, in the long term perspective, the Nordic pulp and paper industry needs to maintain the advantages offered by its worldwide technological leadership by developing value-added products based on new knowledge and technology that are less sensitive to the price of the wood raw material. Wood fibres are already today applied in a wide range of products. In addition to traditional paper products, wood-fibres are also used in for example hygienic products, advanced packaging products and building products as wallboards and MDF boards. The use of wood fibres and their components could, however, be vastly broader than it is at present. One particular promising application of wood fibres is as reinforcement in composite applications. However, such use demands for knowledge beyond present state-of-the-art on the fibre ultrastructure and fibre structure. In addition, new knowledge on the relations between fibre ultrastructure and important parameters such as strength and stiffness will be necessary to understand in a better way. The Woodfibre3D project directly addresses these challenges, and will be an important support for both existing and future advanced developing projects that by this means stimulate to further transforming the Nordic Forest based sector into an even more knowledge based and a research intensive sector.

### 1.1.2 Objectives

*Describe the project objectives.*

The concept of WoodFibre3D is to apply and combine a set of advanced state-of-the-art 3D characterisation facilities to gain increased fundamental competence on the fibre structures at cell-/fibre-/wood-structure level. This competence will be combined with modelling expertise to understand and predict the mechanical properties of the wood fibres and how they can be utilised as reinforcements in composite applications. To reach this vision the project has defined the following scientific objectives;

- Develop and apply sample preparation strategies involving selective staining compatible with Field Emission Transmission Electron Microscopy (FE-TEM) tomography requirements.
- Apply FE-TEM tomography to obtain a documented 3D understanding of wood fibre ultrastructure and composition for a set of fibre materials.

- Apply X-ray microtomography to obtain a documented 3D understanding of
  - wood fibre structure.
  - fibres as reinforcement in a polymer.
- Develop effective software tools to handle and quantify the achieved large 3D data sets.
- Develop finite element models (FEM) to describe relations between fibre ultrastructure – fibre structure – fibre reinforced polymer structure based on the 3D structure data on all three different levels.
- Verify the modelling results with mechanical testing.

## 1.2 Results and discussion

*Main achievements of the project, quality, innovativeness, industrial relevance and contribution to competitiveness, environmental and societal impact.*

The main conclusions and capabilities generated by the projects are described below. In addition, X- $\mu$ CT and complementary electron microscopy techniques (FESEM and TEM) on wood fibre- and wood fibre reinforced composites is a rather novel field with few scientific works available in the literature. A more thorough understanding of wood fibres, composite microstructure and the relations to mechanical properties are essential for industrial success and development. The work on X- $\mu$ CT, the model development and improved image analysis capabilities performed within Woodfibre3D will contribute to this. In this way, the present work also has an indirect environmental impact since the project address competence that benefit further development of renewable and sustainable materials.

## 1.3 Conclusions

*The most important contributions to the state-of-the-art, derived from the results and discussion.*

- New tools (algorithms) to effectively characterize relevant microstructural parameters for 3D images of wood-fibre materials.
- Improved micromechanical models for wood-fibre composites to relate these parameters to important engineering properties: stiffness, strength and dimensional stability.
- Strategies how to implement these tools in materials development have been outlined, e.g. reduce fibre length degradation in injection moulded wood composites.

## 1.4a Capabilities generated by the project

*Knowledge generated in the project / outcomes of the project, such as unpublished doctoral theses, patents and patent applications, computer programs, prototypes, new processes and practices; established new businesses; potential to create new business opportunities in the sector.*

- Algorithms for individual fibre segmentation and fibre network simulation have been developed. Those will be utilised and further developed in future projects. Continued development plan for this technology exists, which will result in multiple publications in the following years.
- Large amount of information regarding X- $\mu$ CT scanning procedures of low-contrast samples has been generated. Scans in special atmospheres have been considered as well. This information will be used in future research on composite materials and in training of new operators for X- $\mu$ CT devices in Jyväskylä and for improvement of image quality in the specific imaging devices.

- Database of X- $\mu$ CT images of wood fibre composite materials has been built. This database contains hundreds of tomographic images of different kinds of fibres and WFCs in varying states, e.g. wet and dry, before and after tensile testing etc. This data will be used in future projects considering similar topics.
- FE-TEM tomography technique has been established. Several methods have been developed for micro- and nanostructural characterisation, based on SEM and FE-SEM. The applied techniques have given a comprehensive description of cross-sectional and surface characteristics of a set of pulp fibres.
- New methodology discovered to stain microfibrillated cellulose for HRTEM analysis.

### 1.4b Utilisation of results

*Give a brief description of how the results of the research and development have been used and/or what is the exploitation plan or plans for transferring the results into practice.*

Woodfibre3D is a rather fundamental research project, and the results of the projects will be utilized in different ways;

- Incorporated in ongoing national projects.
- Included in new initiatives, both national and transnational. The partners have already submitted a new common initiative, where also industrial participation is present.
- A large database of X- $\mu$ CT images of wood fibre composite materials has been built. This could be utilized as a basis for further R&D.

## 1.5 Publications and communication

### a) Scientific publications

*For publications indicate a complete literature reference with all authors and for articles a complete name. Indicate the current stage of the publishing process when mentioning texts accepted for publication or in print. Abstracts are not reported. Indicate the five most important publications with an asterisk.*

#### 1. Articles in international scientific journals with peer review

Miettinen A, Luengo Hendriks CL, Chinga-Carrasco G, Gamstedt EK, Kataja M. A non-destructive X-ray microtomography approach for measuring fibre length in short-fibre composites (2010), Submitted to Composites Science and Technology.

F. Malmberg, J. Lindblad, C. Östlund, K.M. Almgren and E. K. Gamstedt, "Measurement of fibre-fibre contact in three-dimensional images of fibrous materials obtained from X-ray synchrotron microtomography", accepted in *Nuclear Instruments and Methods in Physics Research Section A*, 2010.

Yamakawa, A., Chinga-Carrasco, G.: "Classification of wood fibre cross-sectional shapes", Hybrid Artificial Intelligence Systems, Lecture Notes in Computer Science, Volume 6076/2010, 144-151.

Chinga-Carrasco, G., Johnsen, P.O., Øyaas, K.: "Structural quantification of wood fibre surfaces - morphological effects of pulping and enzymatic treatments". *Micron* 41(6): 648-659.

Luengo Hendriks CL. Constrained and dimensionality-independent path openings. *IEEE Transactions on Image Processing* 19(6):1587-159, 2010

G. Chinga-Carrasco, M. Lenes, P. O. Johnsen, E-L. Hult. Computer-assisted scanning electron microscopy of wood pulp fibres: dimensions and spatial distributions in a polypropylene composite. *Micron*, 2009.

In addition to these accepted manuscripts – a range of manuscripts is in progress.

## **2. Articles in international scientific compilation works and international scientific conference proceedings with peer review**

Yamakawa, A. and Chinga-Carrasco, G.: "Updating the Shape descriptor plugin for automatic classification of TMP fibre cross-sections". 3rd ImageJ User and developer conference. Luxembourg, October 27-29 2010

Yamakawa, A., Chinga-Carrasco, G.: "Classification of wood fibre cross-sectional shapes", 5th International conference on Hybrid Artificial Intelligence Systems, San Sebastian Spain, June 23-25, 2010.

Malmberg, F., Lindblad, J., Östlund, C., Almgren, K.M. and Gamstedt, E.K., "Measuring fibre-fibre contact in 3D images of fibrous materials", *Proceedings of the 13th European Conference of Composite Materials*, Asp, L.E. and Gamstedt, E.K. (Eds.), Stockholm, 2008, 10 p.

Lindström, M., Gamstedt, E.K., Berthold, F., Varna, J. and Wickholm, K., "Hierarchical design as a tool in development of wood-based composite applications", *Proceedings of the 13th European Conference of Composite Materials*, Asp, L.E. and Gamstedt, E.K. (Eds.), Stockholm, 2008, 7 p.

Almgren, K.M, Gamstedt, E.K., Berthold, F. and Lindström, M., "Hygroexpansion of wood-fibre composite materials: Effects of cell-wall cross-linking and composition of thermoplastic matrix", *Proceedings of the 13th European Conference of Composite Materials*, Asp, L.E. and Gamstedt, E.K. (Eds.), Stockholm, 2008, 10 p.

Lindström, M., Berthold, F., Gamstedt, K., Varna, J. and Wickholm, K., "Hierarchical design as a tool in materials development", *Proceedings of the 10<sup>th</sup> International Conference on Progress in Biofibre Plastic Composites*, Toronto, 2008, 7 p.

Tanem, B.S., Li, Y., Gamstedt, K., Vullum, P.E., Stenstad, P. and Walmsley, J.C., "Elastic properties of MFC: Advanced electron microscopy combined with micromechanical modeling", paper #CELL198, *Proceedings of the 237th ACS National Meeting*, Salt Lake City, 2009.

Tanem, B.S., Li, Y., Gamstedt, K., Vullum, P.E., Walmsley, J.C. and Stenstad, P., "Structure and properties of MFC reinforced nanocomposites: Advanced electron microscopy combined with micromechanical modeling", paper #CELL222, *Proceedings of the 237th ACS National Meeting*, Salt Lake City, 2009.

Bjurhager, I., Ljungdahl, J., Wallström, L., Gamstedt, K. and Berglund, L.A., "Effects of polyethylene glycol treatment on the mechanical treatment of oak – A model study for better understanding of the Vasa ship", *Proceedings of the COST Action FP0802 Annual Conference on Experimental and Computational Methods in Wood Micromechanics*, Hofstetter, K. (Ed.), Vienna, 2009, 51-52.

Neagu, R.C., Gamstedt, E.K. and Bardage, S.L., "Analytical micromechanical analysis of the hygroelastic behaviour of compression wood tracheids", *Proceedings of the COST Action FP0802 Annual Conference on Experimental and Computational Methods in Wood Micromechanics*, Hofstetter, K. (Ed.), Vienna, 2009, 85-86.

Tanem, B.S., Li, Y., Vullum, P.E., Gamstedt, E. K. Relations between structure and elastic properties of microfibrillated cellulose based on wood, *Proceeding of the 239th American Chemical Society National Meeting & Exposition*, San Francisco, CA, March 21-25, 2010.

Axelsson M. Estimating 3D fibre orientation in volume images. 19th International Conference on Pattern Recognition.

Axelsson M (2009). An Evaluation of Scale and Noise Sensitivity of Fibre Orientation Estimation in Volume Images. 15th International Conference on Image Analysis and Processing.

Wernersson E, Luengo Hendriks CL, Brun A (2009). Generating synthetic  $\mu$ CT images of wood fibre materials. 6th International Symposium on Image and Signal Processing and Analysis.

Wernersson E, Brun A, Luengo Hendriks CL (2009). Segmentation of Wood Fibres in 3D CT Images Using Graph Cuts. 15th International Conference on Image Analysis and Processing.

### **3. Articles in national scientific journals with peer review**

[Text]

### **4. Articles in national scientific compilation works and national scientific conference proceedings with peer review**

[Text]

### **5. Scientific monographs**

F. Mangiapane, MSc thesis, 2009, Department of fibre and polymer technology, KTH, "Moisture expansion of wood-fibre reinforced composite: Micromechanics and microstructural characterization"

T. Joffre, MSc thesis, 2010, Department of fibre and polymer technology, KTH, "Modelling of fibre swelling due to moisture: Effects of constraining matrix in composite materials"

A. Miettinen, MSc thesis, 2009, Department of Physics, University of Jyväskylä, "Kuituverkostojen yksityiskohtainen analysointi korkean resoluution kolmiulotteisista kuvista" (Detailed analysis of fibre networks using high-resolution three-dimensional images)

### **6. Other scientific publications, such as articles in scientific non-refereed journals and publications in university and institute series**

[Text]

#### **a) Other dissemination**

*Such as text books, manuals, user guidelines, newspaper articles, TV and radio programmes, meetings and contacts for users and results. Dissemination of results to industrial partners and industrial partners dissemination within the company.*

Chinga-Carrasco G, Miettinen A, Luengo Hendriks CL, Gamstedt EK, (2011) Structural characterisation of wood pulp fibres and their nanofibrillated materials for biodegradable composite applications. Invited book chapter contribution. Composite Materials / Book 3", ISBN 978-953-307-1099-2

Axelsson M (2008). Tracking tubular structures in volume image data. Swedish Symposium on Image Analysis.

Malmberg F, Östlund C, Borgefors G (2008). Graph cut based segmentation of phase contrast volume images of fibrous materials. Swedish Symposium on Image Analysis.

Wernersson E, Brun A, Luengo Hendriks CL (2009). Closing Pores and Segmenting Individual Fibres in 3D Images of Wood Fibre Composites using Curvature Information and Graph Cuts. Swedish Symposium on Image Analysis.

Luengo Hendriks CL (2010), Path openings and their applications. Swedish Symposium on Image Analysis.

Miettinen A., Koivu V., Turpeinen T. and Kataja, M., An automated method for recognizing individual fibres from 3D tomographic images, Poster presentation at COST action FP0802 workshop "Single fibre testing and modelling", Innventia, Stockholm, Sweden, 4-5 Nov 2009

**Best poster award** at the COST Action FP0802 workshop on Wood Micromechanics in Hamburg, October 2010: "Effects of matrix constrain on swelling of wood fibres in composite materials", Thomas Joffre, Kristofer Gamstedt, Cris Luengo, Erik Wernersson.

Mori Hult E. L., Tanem BS, WoodFiber3D; Structure-property relations of wood fibres – 3D characterisation and modelling, *presentation at Programme opening seminar at WoodWisdom-Net*, Berlin, 12 Feb 2008.

Tanem, BS, *Presentation at Research Seminar: Recent advances in fibrillar nanocellulose research – Characterisation and applications*. Trondheim, 12 Nov. 2008.

Tanem, B.S., Li, Y, Vullum, P.E., Gamstedt, E. K, C. L.L Hendriks, Elastic properties of microfibrillated cellulose and its nanocomposites; Advanced electron microscopy combined with micromechanical modelling, *Oral presentation at the Nanomat conference*, Lillehammer, 16-17th of June

Tanem, BS , *WoodFibre3D Lecture at WoodWisdom -Net Mid-term Seminar*, Stockholm, 11 Nov, 2009.

Chinga-Carrasco, G, Tanem BS, Multiscale structural characterisation of wood fibre-reinforced composites. *Poster presentation at WoodWisdom -Net Mid-term Seminar*, Stockholm, 11 Nov, 2009.

E. K. Gamstedt, Estimation of Stiffness of Microfibrillated Cellulose based on Nanostructure Characterized by Transmission Electron Microscopy , *Lecture at 6th International ECNP Conference on Nanostructured polymers and nanocomposites*, Madrid, 28-29<sup>th</sup> of April 2010.

## 1.6 National and international cooperation

*Give a brief description of the cooperation/ networking (partnership between the project participants and how this has developed; industrial involvement; synergies of industrial and research expertise; Has the project collaborated with similar projects in the WW-Net countries or other regions, or established new links with/ between local or international organisations involved in the respective research field? Describe how these partnerships have supported the project. National vs. transnational aspects in the project; added value for the project and its impacts which result from transnational cooperation.*

Most of the participants had already contact/interactions through separate projects/cooperation at the time when WoodFibre3D was launched. However, the partnership within WoodFibre3D has gradually developed through active use of common meetings (three common meetings in 2008, three meetings in 2009, three meetings in 2010 – in addition to several telephone conferences and more informal and dedicated meetings involving only a few partners). We used these meetings to get better known to each other (competence, lab facilities etc) and identification of common goals and scientific challenges. We have tried to put emphasise on perusing challenges that require a genuine cross-disciplinary approach, and therefore have to involve several partners. Student exchange has been another positive mean to enhance collaboration.

Since Woodfibre3D is a rather fundamental research project, no industrial participant has been directly involved in the work. However, the partners submitted a new common proposal to Woodwisdom-net in Dec 2010, where industry is involved, and one of the purposes is to utilize the rather fundamental knowledge acquired in Woodfibre3D to solve more industrial-oriented challenges.

There has not been any collaboration with other WW-net projects. However, contact has been established with a cost action, where one of the partners (KTH) in Woodfibre3D is a key partner. The mid-term seminar in WoodWisdom arranged in Stockholm in Nov2009 was a good arena and opportunity for networking and exchange of experience. We have had discussions with the people at the TOMCAT beamline at the Paul Scherrer Institute to further develop our tools for simulating image acquisition and develop more advanced imaging protocols. In particular they have been very helpful during the writing of a paper. We have also been offered to visit them for a longer time to extend the collaboration on image analysis and acquisition of wood fibre images.

Most of the partners in Woodfibre3D have had ongoing complementary national activities, which have been very beneficial also for the Woodfibre3D activities, in terms of experience, choice of materials, experimental procedures etc.