Observing anatomical modifications of wood during thermal treatment with ESEM

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ABSTRACT

ESEM- (Environmental Scanning Electron Microscope) equipped with an hot chamber operating at low vacuum conditions was used to observe the kinetic of change of wood structure of Spruce samples due to thermal degradation. The device allowed to control the temperature in the chamber and to heat up the sample (by conduction) according to a desired temperature ramp. HR pictures of the wood structure at a magnification of 1200 X were taken at regular interval during all the thermal process. The paper reports results of some preliminary observations performed on cross section of Spruce samples during a thermal exposition from environmental T up to 390 °C. Post image analysis allowed to quantify the modification rate of some selected wood structure parameters such as cell wall thickness and behaviour of fracture propagation.

INTRODUCTION

Considerable studies have been performed on changes in structure of heat treated woods, many of them based on the observation of changes occurred as a consequence of the treatment (Awoyemi 2011, Biziks 2013). Since it is nearly impossible to observe the same microscopic wood elements before and after the process, such studies are necessarily based on a statistical approach to an average change of some anatomical parameters such as the dimension of lumens or wall thickness. Several factors influence the microscopic observation and the results:

- the cutting of the samples tends to deforms the wood cells at the cutting surface and, above all when the wood is treated, produces cracks on the cell walls;
- the effect of the preparation of the sample, such as dehydration and the metal coating might produces some unrealistic behaviour of wood during the pyrolysis.

Finally, a "before and after" approach does not allow to observe what happens during the thermal process.

In this paper the first experiences on the use of ESEM- (Environmental Scanning Electron Microscope) for the observation of structural changes of small samples of spruce during thermal treatment in low vacuum condition are reported. The ESEM allows to operate with wood samples at an initial MC at the equilibrium with the environment (about 12%) and without any metal coating. It is equipped with a hot chamber that allows to control the temperature profile during time. It operates at low vacuum (3.15 mbar).

In this condition is possible to replicate in small scale the thermal treatment conditions of the TERMOVUOTO process were the oxygen inside of the reactor is substituted by partial vacuum. The Thermal Process under Vacuum have been developed and studied in this last years (Allegretti 2012, Ferrari 2013) with results indicating some improving in the residual mechanical properties of the treated wood compared to others technologies (Candelier 2013). The main difference between the condition in ESEM and

TERMOVUOTO is the level of vacuum which, in the latter is roughly 100 time higher (about 200 mbar in normal condition).

EXPERIMENTAL

Small samples of Norway spruce (Picea abies Karst.) 3 x 3 x 3 mm have been cut by hand blade from different dried sawn board. No water, or other products have been used to soften the wood before cut. After cutting they have been equilibrated at 12% MC.
The experimental setup was configured to observe the cross section of the specimens at a X 1200 constant magnification.
Three experiments have been performed Temperature increase was fixed at 2 °C/min from the environmental T up to 390 °C. With this set up the duration of each tests was about 3 hours. Pressure had been kept constant during all the experiment at 3.15 mbar.
Pictures have been taken at temperature steps of 50 °C.
Post-image analysis was performed measuring the variation of size and shape of some selected anatomical elements as depicted in Figure 1 and Table 1.

Table 1: The wood-anatomical characteristics observed during the thermal treatment.

Code	Wood element
1	Latewood tangential cell wall (thickness)
2	Latewood radial cell wall (thickness)
3	Earlywood tangential cell wall (thickness)
4	Earlywood radial cell wall (thickness)
5	Splits
6	Parenchymatic ray
7	Earlywood radial lumen
8	Earlywood tangential lumen
9	Latewood tangential lumen
10	Latewood radial lumen

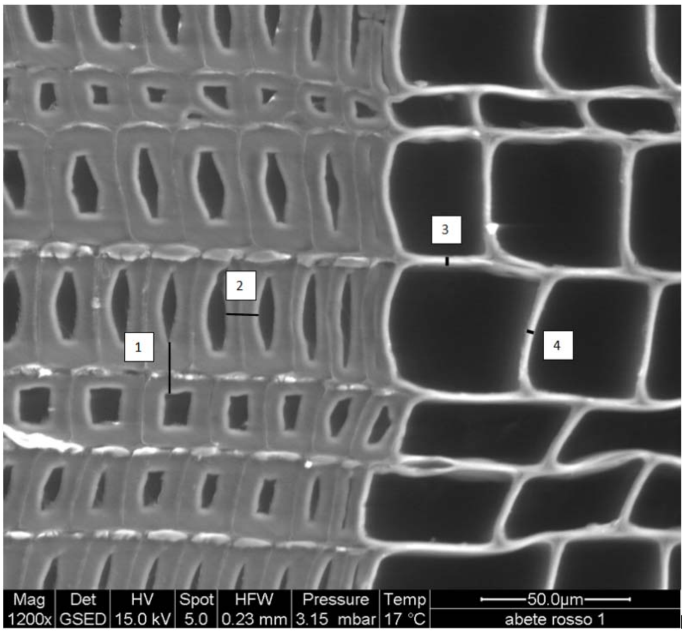

Figure 1: An example of the analysed wood parameters.

RESULTS AND DISCUSSION

Despite some problems not solved yet such as:

- sharpness of images, not so high as for SEM;
- deformation of images probably due to the image scanning system and/or to the roto-translation of the plan of specimen due deformations;
- the ESEM allowed to make some interesting observation.

Cell wall

The cell wall showed a typical pattern of deformation, in general repeatable in different tests and in different selected elements. It characterised by:

1. a first slight swelling stage up to about 100 °C. Our explanation of the phenomena is the pressure exerted by water vapour on the wood walls which started acting at low temperature since the vacuum was applied in the chamber and until the total evaporation of water. A related phenomena might be the development of blisters observed on the transversal surface of the wood walls, mainly at level of S2 layer, from a temperature of 110 °C (Figure 2). This temperature roughly corresponds to the glass transition temperature of lignin in softwood;

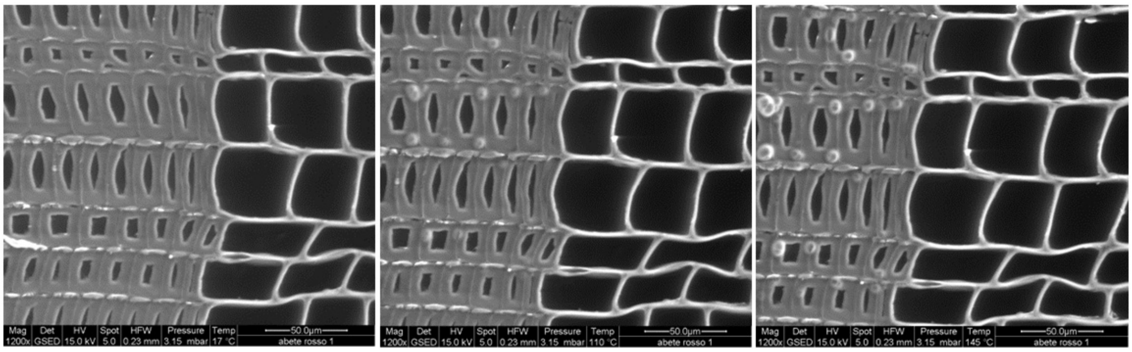

Figure 2: Development of blisters on the transverse surface of the wood. Pictures at 20°, 110° and 145 °C.

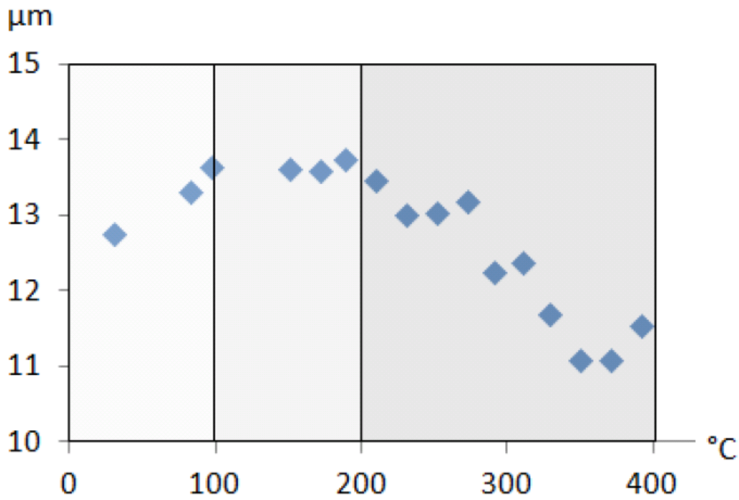

Figure 3: % variation of the cell wall thickness of sample 2.

2. a second stage in the T range between 100 °C and 200 °C without significant dimensional variation;
3. a third stage from 200 °C up to the final T with a significant wall shrinkage. At the end of the experiments shrinkages in the range between 19% and 39% were observed (Figure 3).

ANOVA analysis showed no significant differences of shrinkages between early and late wood and between radial and tangential direction. However in this case, the problems above described could affect the quality of data, above all for the small size elements such as the early wood walls and the membranes pits.

Cell lumen

The area of the cell lumens seemed to decrease (10% average). However data were often contradictory or not clear. The cell lumens were often affected by crushes, collapses and strong deformations. In some cases (Figure 4) the lumen size increased with the temperature. It is clear that the effect of mechanical forces exerted by the deformation of the surrounding structure produces a *Poisson effect* and in general deformation on the single cells.

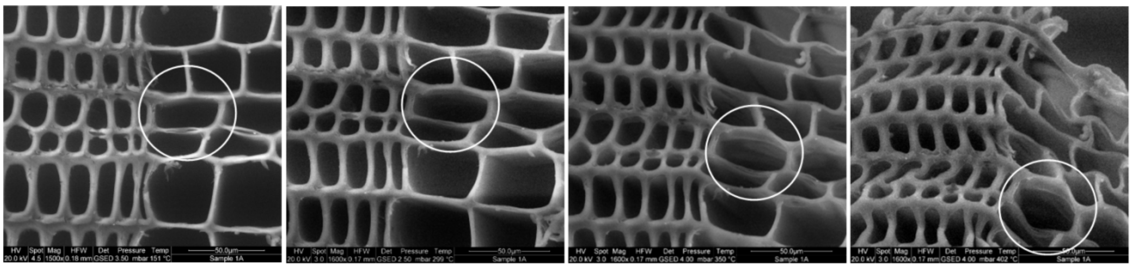

Figure 4: The increase of temperature resulted in the curling, rotation and tilt of the sample. Pictures at 150, 300, 350 and 400 °C.

Splits

The occurrence of ex-novo splits was rarely observed. The 3-D organisation of the wall structure allowed to contrast the stress without fracture (Figure 5). On the other hand, it was frequently observed that the stress tended to discharge on the splits already existing between the cells, with a huge increase their gap.

Other elements

The measures of the others observed elements (Parenchymatic rays, structure of pits), have not provided relevant results.

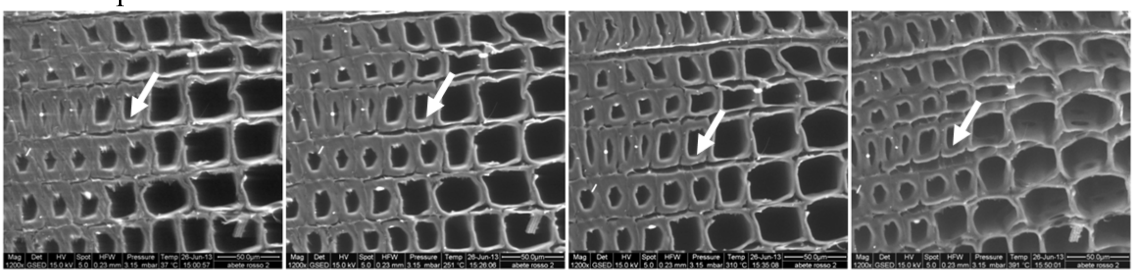

Figure 5: The same sample is photographed at 37, 251, 310 and 391 °C. The cell wall thickness is reducing while gaps between the elements are increasing. The last picture on the right is showing the beginning of the sample curling.

CONCLUSIONS

This work explored the potentialities of ESEM to investigate the structural change of wood during the process of thermal modification. The methodology especially matches with thermal treatment systems using vacuum as protective atmosphere such as the TERMOVUOTO technology.
Some qualitative interesting observation have been here described, such as the development of blister in the S2 layer of the wall cell occurring from about 100 °C as a consequence of the vapour pressure on the wall cell and the lignin plasticisation.
This methodology might be also an useful tool to observe and to explain the dynamic of fractures which strongly affect the quality of the thermal treatment in some species such as oak and black locust.
Next steps will be the improvement of the methodology, concerning the image acquisition and the image analysis. Further experiments are planned on other species, softwood and hardwood.

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